Town of Islip

Interim Site Management Plan

Long Island MacArthur Airport

NYSDEC Site 152251

June 4, 2026

Interim Site Management Plan

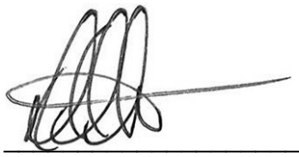
Long Island MacArthur Airport, NYSDEC Site# 152251

June 4, 2026

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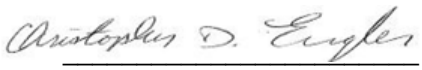
Appendix F. **Community Air Monitoring Plan (CAMP)**

Acronyms and Abbreviations

AFFF	Aqueous Film Forming Foam
Arcadis	Arcadis of New York, Inc.
AOCs	Areas of concern(s)
CAMP	Community Air Monitoring Plan
CFR	Code of Federal Regulations
COCs	contaminants of concern
COU	Change of use
DER-10	Technical Guidance for Site Investigation and Remediation
DER-31	Green Remediation
FAA	Federal Aviation Administration
FBOs	fixed-based operators
HASP	health and safety plan
Ng/L	Nanograms per liter
NYSDEC or Department	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
NYSDOT	New York State Department of Transportation
PE	Professional Engineer
PFAS	polyfluoroalkyl substances
PFOS	perfluorooctane sulfonate
PFOA	perfluorooctanoic acid
QAPP	Quality Assurance Project Plan
QEP	qualified environmental professional
RAOs	Remedial Action Objectives
SCOs	Soil Cleanup Objectives
SMP	Site Management Plan
SPLP	synthetic precipitation leaching procedure

Professional Engineer Certification

I CHRISTOPHER ENGLER, certify that I am currently a NYS registered professional engineer and that this Interim Site Management Plan was prepared in accordance with all applicable statutes and regulations and in substantial conformance with the DER Technical Guidance for Site Investigation and Remediation (DER-10) and Green Remediation (DER-31).


_____ [P.E.]

_____06/04/2026____Date

1 Introduction

Arcadis of New York, Inc. (Arcadis) prepared this Interim Site Management Plan (ISMP) on behalf of Town of Islip for Long Island MacArthur Airport located in Ronkonkoma, New York (hereinafter referred to as the Site; see **Figure 1**). The Site is listed by the NYSDEC as a Class 2 Inactive Hazardous Waste Disposal Site Remedial Program, Site No. 152251, which is administered by New York State Department of Environmental Conservation (NYSDEC or the Department). This ISMP was prepared in accordance with the requirements of the NYSDEC's Technical Guidance for Site Investigation and Remediation (DER-10), dated May 2010, and the guidelines provided by the NYSDEC.

This ISMP will address soil management at the Site and is primarily focused on per- and polyfluoroalkyl substances (PFAS), including perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS), but also applies to other potential contaminants of concern, including Volatile Organic Compounds (VOCs) and Semi-Volatile Organic Compounds (SVOCs).

2 Site Description

The Site is located at 100 Arrival Avenue in the Hamlet of Ronkonkoma, Town of Islip, New York (**Figure 1**). The Site consists of a complex of buildings and structures positioned on approximately 1,141.8 acres. The Site is used as a Federal Aviation Administration (FAA) Part 139 airport that serves commercial and general aviation flights. The Site is bounded to the north by the Long Island Railroad facility and Railroad Avenue, to the west by Smithtown Avenue with mixed commercial and industrial use, to the east by Lincoln Avenue with mixed residential and commercial use, and to the south by Veterans Memorial Highway with mixed commercial and industrial use. The owner of the Site is the Town of Islip.

Currently, the Site comprises a runway/taxiway system, airport terminals, commercial aviation buildings, and airport service buildings. In general, MacArthur Airport operations can be subdivided into core and non-core functions. Core operations can be defined as operations essential to the function of the airport. Core operations include fixed-based operators (FBOs), and non-core operations include concessions/support tenants and satellite areas. Concessions/support tenants include Avis/Budget and Enterprise/National Rental Car, Clarion Hotel, and Swissport Fuel. The Central Aviation Marine Corporation (CAMCO) parcel at 2125 Smithtown Avenue (NYSDEC Site 152206), and the Army National Guard AASF#1 facility are considered separately regulated facilities and are not included within the Site boundary. Additional details on site use were provided in the NYSDEC-approved Record Search Report (Arcadis 2019).

The main contaminants of concern (COCs) are PFAS. PFAS has been detected in soil and groundwater on site; however, PFAS impacts on site have not yet been delineated. A Record of Decision Document has not been prepared for the Site, and Remedial Action Objectives (RAOs) have not been established, however the Site Characterization Report (Arcadis 2022) identified eight Area's of Concern (AOCs) (see Table 2-1 below for details) that will be retained for the Remedial Investigation for the Site. There were other Area's investigated in the SC, however these were not identified as AOCs. The AOCs and other Area's requested by NYSDEC for further investigation are listed below and shown on **Figure 2**. PFAS results from the Site Characterization Report (Arcadis 2022) are shown on Figure 2 through 11.

Table 2-1 Areas Of Concern Summary

Area 1: Recharge Basin #1	Not considered release area resulting from storage and/or use of PFAS-containing materials but may have facilitated migration of PFAS through stormwater runoff conveyed overland to recharge basin. Trace detections of PFAS in sediment and stormwater not indicative of local release. Other parameters not detected or did not exceed applicable criteria.
Area 2: Recharge Basin #2	Not considered release area resulting from storage and/or use of PFAS-containing materials but may have facilitated migration of PFAS through stormwater runoff conveyed overland to recharge basin. Trace detections of PFAS in sediment and stormwater not indicative of local release. Other parameters not detected or did not exceed applicable criteria.
Area 3: Fire Training Area #1: Northeast Wooded Area	Records and field observations indicate historical AFFF use. Highest total PFAS concentration in groundwater is 44,638 ng/L. Total PFAS, along with individual constituents, indicate local release.
Area 4: Fire Training Area #2: Northwest, Behind Compost Facility	Records and field observations indicate historical and current AFFF use. Highest total PFAS concentration in groundwater is 19,754 ng/L. Total PFAS, along with individual constituents, indicate local release.
Area 5: Equipment Cleanout Area by Suffolk County Water Authority Tower	Records indicate current and ongoing AFFF use. Highest total PFAS concentration in groundwater is 1,907 ng/L. Total PFAS concentration, along with individual constituents, indicate local release.
Area 6: Old Fire House	Records and field observations indicate historical AFFF use. Highest total PFAS concentration in groundwater is 67,355 ng/L. Total PFAS concentration, along with individual constituents, indicates local release. PFAS were highest detected during the SC.
Area 8: Glycol Treatment Plant	Total PFAS concentration in groundwater is 6,139 ng/L. Total PFAS concentrations, along with individual constituents detected, indicate a local release. This table gives rationale for why AOCs were retained for PFAS investigation and 1,4-dioxane was not retained as a COC based on the results from SC.
Area 11: Hertz/Avis/Budget Hertz (Northern Parcel) Budget (Southern Parcel)	Records and field observations indicate historical and current use of surfactants and other automotive-related substances. Area is used by Hertz, Budget and other rental car companies. Highest total PFAS concentration in groundwater is 1,213 ng/L. Total PFAS, along with individual constituent concentrations, indicate local release.

This ISMP includes the following appendices that should be referenced and developed on a project-specific basis when planning for intrusive work at the Site:

- Appendix A – Site Contacts
- Appendix B – Field Sampling Plan (FSP)
- Appendix C – Quality Assurance Project Plan (QAPP)
- Appendix D – Request To Import/Reuse Fill Material Form
- Appendix E – Best Management Practice Plan
- Appendix F – Community Air Monitoring Plan (CAMP)

3 Notifications

Notifications are required when ground intrusive activities are conducted at the Site. Soil on site will be managed as potentially contaminated with PFAS for purposes of implementing this ISMP.

Notifications will be submitted to the NYSDEC and the New York State Department of Health (NYSDOH), as needed, in accordance with NYSDEC's DER–10 to the contacts listed in Appendix A. As discussed in the meeting between NYSDEC and The Town of Islip on February 3, 2026, NYSDEC acknowledged that Federal Aviation Administration (FAA) regulations may require priority actions be taken by the Site, meaning the required notification period may not be achieved in all instances of soil excavation. In some instances, the full 60-day period for Change of Use (COU) notification may not always be met; however, NYSDEC will be notified as soon as possible for all FAA priority projects. Priority actions taken by the Site include FAA mandated projects pertaining to safety, security, efficiency and operations. Examples of FAA priority projects can include (but are not limited to) the following:

- Runway and taxiway reconstruction and rehabilitation
- Hot spot mitigation
- Safety area or security improvements
- Hangar or building construction, re-habilitation, or demolition
- Projects that may prevent imminent danger or safety of the public
- Public utility emergency
- Other instances where priority action must be taken to ensure safe and consistent airport operations

A Hot Spot is defined as a location on an airport movement area with a history of collisions or runway incursions, or where pilots and vehicle drivers are at a higher risk of such events due to complex geometry, traffic flow, or visibility restrictions. Events such as these are at the forefront of national safety issues. Runway and taxi resurfacing, hot spot mitigation and safety area improvements have previously been carried out at the site and are necessary to address documented operational hazards, fulfill FAA recommendations, and support the Airport's obligation to maintain a safe and compliant airfield.

Subject to the caveats set forth herein, including where full notice may not be met:

Notification by ISP to the NYSDEC and the NYSDOH will be required for the following reasons:

1. 60-day advance notice of any proposed changes in site use, also referred to herein as "Change of Use" (COU), that are required under the terms of the Order on Consent, 6 NYCRR Part 375 and/or

Environmental Conservation Law which establishes New York State's regulatory framework for investigating and remediating contaminated sites through programs such as the Brownfield Cleanup Program, State Superfund, and Environmental Restoration Program.

2. Seven-day advance notice of any field activity associated with the Remedial Investigation (the RI/FS Work Plan is still in development by ISP).
3. 15-day advance notice of any proposed ground-intrusive activity pursuant to the ISMP including upgrades or repairs to buried infrastructure or other structural repairs that disturb site soils. If the ground-intrusive activity qualifies as a change of use as defined above, the above-mentioned 60-day COU notice is also required.
4. Notice within 48 hours, as is possible, of structural repairs or upgrades to buried infrastructure and utilities and a COU will also be submitted as appropriate
5. Verbal notice by noon of the following day of any emergency, such as a fire, flood, or earthquake, that reduces or has the potential to reduce the effectiveness of engineering controls in place at the Site, with written confirmation within 7 days that includes a summary of actions taken, or to be taken, and the potential impact to the environment and the public.
6. Follow-up status reports on actions taken to respond to any emergency event requiring ongoing responsive action submitted to the NYSDEC within 45 days describing and documenting actions taken to restore the effectiveness of the ECs.

Notifications from ISP to the NYSDEC and the NYSDOH project manager will include:

- A description of the work to be performed consisting of the location and areal extent of excavation, plans/drawings for site re-grading, intrusive elements or utilities to be installed under the soil cover, estimated volumes of contaminated soil to be excavated, any modifications of truck routes, and work that may impact an engineering control;
- Identification if soil being removed is planned to be backfilled to the excavation it came from, or if proposed for reuse onsite and where is it proposed to be reused onsite.
- If excavated soil is from an AOC and is being proposed for reuse onsite, it will need to be analyzed prior to being used anywhere outside of the AOC.
- A summary of environmental conditions anticipated to be encountered in the work areas including the nature and concentration levels of COCs, potential presence of grossly contaminated media, and plans for any pre-construction sampling;
- A schedule for the work, detailing the start and completion of intrusive work, and submittals (e.g., reports) to the NYSDEC documenting the completed intrusive work;
    - A summary of the applicable components of this ISMP;
    - A statement that the work will be performed in compliance with this SMP, 29 Code of Federal Regulations (CFR) 1910.120 and 29 CFR 1926 Subpart P which establishes excavation and trenching safety standards to prevent cave-ins and protect workers in construction excavations and protect workers in construction excavations.
    - A copy of the contractor's health and safety plan (HASP), in electronic format.
    - Identification of disposal facilities for potential waste streams (as applicable); and
    - Identification of sources of any anticipated backfill, along with the required request to import form and supporting documentation including, but not limited to, chemical testing results (as applicable).

The NYSDEC and the NYSDOH project manager will review the notification and may impose additional requirements for the excavation that are not listed in this ISMP.

4 Soil Management Plan

Community air monitoring for particulates will be required during soil disturbance activities, including excavation, backfilling, regrading, or any other activity involving reworking of native soils. When work areas within known Areas of Concern (AOCs) (as determined in the Site Characterization Report, Arcadis 2022) are within 20 feet of potentially exposed populations or occupied structures, the continuous monitoring locations for particulates must reflect the nearest potentially exposed individuals and the locations of ventilation system intakes for nearby structures. VOC monitoring will only be conducted if working in an area where hydrocarbons or other VOCs could potentially be present. The use of engineering controls, such as vapor/dust barriers, temporary negative-pressure enclosures, or special ventilation devices, should be considered to prevent exposures related to the work and to control dust and odors. Consider implementing the planned activities when potentially exposed populations are at a minimum such as during weekends or evening hours in non-residential settings.

If total particulate concentrations opposite the walls of occupied structures or next to intake vents exceed 150 micrograms per cubic meter, work should be suspended until controls are implemented and successfully reduce the total particulate concentration to 150 micrograms per cubic meter or less at the monitoring point as per the CAMP **(Appendix F).**

4.1 Soil Screening and Reuse Planning

Soil screening will be conducted any time soil disturbance of potentially contaminated soil occurs within an AOC, Soil screening will be performed by a qualified environmental professional (QEP), as defined in 6 NYCRR Part 375, a Professional Engineer who is licensed and registered in New York State (PE), or their properly trained designee. Soil screening will consist of inspections of soil for visual or olfactory signs of contamination. If working in an area where VOCs are a suspected contaminant of concern, then a PID will also be used for screening.

If working outside of an AOC, soil screening will not be required, however CAMP monitoring will be performed as per Section 4.10. Within an AOC, soil screening will be performed by a QEP, as defined in 6 NYCRR Part 375, a PE, or their properly trained designee.

Site work involving soil disturbance will attempt to avoid known AOCs, to the extent possible. PFAS cannot be identified via field soil screening methods, however if unexpected indications of impact are identified, work will be temporarily stopped and steps listed in Section 4.9 of this ISMP will be followed. The QEP, PE or their properly trained designee will ensure that procedures defined for materials reuse in this ISMP are followed and that deleterious material encountered during project earthwork activities (i.e., visually exhibiting indications of impact via odor or staining) will be disposed offsite in accordance with Sections 4.3 and 4.4 of this ISMP. The intent of the ISMP is not to investigate and delineate deleterious material encountered during earthwork activities. In areas where hydrocarbons, or other volatile organics could be reasonably anticipated (e.g. during underground storage tank removal), a photoionization detector (PID) will be kept onsite and used to screen soil. A QEP, PE, or their properly trained designee will perform the screening.

The NYSDEC will be notified of anticipated reuse of soil. If excavated soil is from an AOC and is being proposed for reuse onsite, it will need to be sampled for PFAS prior to being used anywhere outside of the AOC. Soil outside of AOCs may be reused onsite without analytical testing, however NYSDEC must be notified of the plan

for reuse. Soil excavated from an AOC can be reused within the same general area of the excavation without sampling for PFAS.

The sampling frequency for reuse will be in accordance with DER-10 Table 5.4(e)10 which is a table within the New York State Department of Environmental Conservation's DER-10 Technical Guidance for Site Investigation and Remediation. It is part of Section 5.4(e), which establishes the sampling, testing, and acceptance criteria for soil or fill material proposed for import or reuse at a remediation site unless previous approval is obtained from the NYSDEC project manager for modification of the sampling frequency. The analytical results of soil/fill material testing must meet the Site use criteria presented in NYSDEC DER-10 Appendix 5 – Allowable Constituent Levels for Imported Fill or Soil for constituents listed and the NYSDEC Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances (April 2023) guidance values. Approvals for modifications to the analytical parameters must be obtained from the NYSDEC project manager before the sampling event.

Soil from an AOC proposed for reuse elsewhere onsite is required to meet the Protection of Groundwater Use Guidance Values for PFOA and PFOS due to groundwater contamination onsite. Unrestricted Use Soil Cleanup Objectives (SCO) also apply as an environmental easement is not yet in place on the site.

Soil/fill material for reuse on site will be segregated and staged on site. Anticipated sizes and locations of stockpiles will be provided in the 15-day notification to the NYSDEC project manager. Stockpile locations will be based on the locations of site excavations and proximity to nearby site features. Material reuse on site will comply with requirements of NYSDEC DER-10 Section 5.4(e)4 which refers to a requirement within DER-10's soil/fill import evaluation rules that outlines part of the criteria and procedures for assessing whether imported or reused soil meets the standards established in Section 5.4(e) for acceptable fill material as indicated in the QAPP **(Appendix C)**.

When planning for soil reuse, soil sampling analytical results from the laboratory will be provided in a Category B Report, with a data usability summary report (DUSR) and Electronic Data Deliverables (EDDs – in EQUiS-compatible format) to be provided at the conclusion of the project.

Any modifications to the requirements of DER-10 Section 5.4(e)4 must be approved by the NYSDEC project manager.

4.2 Soil Staging

Soil stockpiles will be continuously contained within a berm and/or silt fence. Hay bales will be used as needed near catch basins, surface waters, and other discharge points.

Stockpiles will be kept covered at all times with appropriately anchored tarps. Stockpiles will be routinely inspected, and damaged tarp covers will be promptly replaced.

Stockpiles will be inspected, at a minimum, once each week and after every storm event. Results of inspections will be recorded in a logbook and maintained at the Site available for inspection by the NYSDEC.

4.3 Materials Excavation and Load Out

A QEP, PE, or their properly trained designee will oversee invasive work, excavation and load-out of excavated material.

The owner of the property and remedial party (if applicable) and its contractors are responsible for safe execution of invasive and other work performed under this Plan.

The presence of utilities and easements on the Site will be investigated by the QEP, a PE, or their properly trained designee. It will be determined whether a risk or impediment to the planned work under this ISMP is posed by utilities or easements on the Site. A site utility markout will be completed for utilities before any ground-intrusive activities at the Site by the performing contractor.

Trucks transporting contaminated soil leaving the Site will be appropriately lined, tarped, securely covered, manifested, and placarded in accordance with appropriate federal, state, local, and New York State Department of Transportation (NYSDOT) requirements (and all other applicable transportation requirements). Trucks transporting contaminated soil must have either tight-fitting opaque covers that are secured on the sides and/or back or opaque covers that are locked on all sides.

A truck wash will be operated on site for trucks working within an AOC or within an area where contamination is discovered. The QEP or a PE (or their properly trained designee) will be responsible for ensuring that outbound trucks that have come into contact with contaminated soil will be washed at the truck wash before leaving the Site until activities performed under this section are complete. Truck wash waters will be collected and disposed of off-site in an appropriate manner.

Locations at which vehicles enter or exit the Site will be inspected daily for evidence of off-site soil tracking.

The QEP, PE, or their properly trained designee will be responsible for ensuring that all egress points for truck and equipment transport from the Site are clean of dirt and other materials derived from the Site during intrusive excavation. Adjacent streets will be cleaned as needed to maintain a clean condition with respect to site-derived materials. Material accumulated from the street cleaning and egress cleaning will be disposed of off-site at a permitted landfill facility in accordance with applicable local, state, and federal regulations.

4.4 Backfill from Off-Site Sources

Materials proposed for import onto the Site will be approved by the QEP, PE, or their properly trained designee and will comply with provisions in this ISMP before receipt at the Site. A Request to Import/Reuse Fill or Soil form, which can be found at http://www.dec.ny.gov/regulations/67386.html, will be prepared and submitted to the NYSDEC project manager allowing a minimum of 5 business days for review. A copy of the form is presented in **Appendix D**.

Material from industrial sites, spill sites, other environmental remediation sites, or potentially contaminated sites will not be imported to the Site.Virgin quarry materials or other materials that qualify for sampling exemption under DER-10 Section 5.4(e)5 will not be sampled prior to import.

Proposed materials for backfilling from off-site sources must be sampled for full suite analytical parameters (PFAS, 1,4-dioxane, volatile organic compounds, semi-volatile organic compounds, metals, polychlorinated biphenyls, pesticides, and herbicides). Analytical results for imported soil requires Category B deliverables and EDDs / EQuIS, but do not require DUSRs. The sampling frequency will be in accordance with DER-10 Table 5.4(e)10 unless previous approval is obtained from the NYSDEC project manager for modification or waiver of the sampling frequency.

Imported soils will meet the backfill soil quality standards established in 6 NYCRR 375-6.7(d) and DER-10 for Unrestricted Use SCOs and for contaminants of concern such as PFOS and PFOA, soil will meet Protection of Groundwater SCOs. Soils that meet 'general' fill requirements under 6 NYCRR Part 360.13, but do not meet backfill objectives for this Site, will not be imported onto the Site without previous approval by the NYSDEC project manager.

If PFOA or PFOS is detected in any sample at a concentration that is at or above the guidance values, then the source of backfill should be rejected unless a site-specific exemption is provided by DER based on results from synthetic precipitation leaching procedure (SPLP) testing, for example. If the concentrations of PFOA and PFOS in leachate are at or above the ambient water quality guidance values for groundwater, then the soil is not acceptable.

PFOA and PFOS are considered semi-volatile compounds; therefore, composite samples are appropriate for these compounds when sampling in accordance with DER-10, Table 5.4(e)10. Laboratory deliverables should be submitted for backfill soil samples in accordance with the QAPP **(Appendix C)**.

Trucks entering the Site with imported soils will be securely covered with tight-fitting covers. Imported soils will be stockpiled separately from excavated materials and covered to prevent dust releases.

4.5 Materials Off-Site Transportation and Disposal

The airport will be responsible for characterizing and disposing of materials at an approved facility, however they may elect to have the performing contractor collect waste characterization samples and submit the results to the airport for review and their use in managing offsite disposal of materials.

Materials will be transported by licensed haulers in accordance with appropriate local, state, and federal regulations including 6 NYCRR Part 364. Haulers will be appropriately licensed and trucks properly placarded.

Material transported by trucks exiting the Site will be secured with either tight-fitting opaque covers that are secured on the sides and/or back or opaque covers that are locked on all sides. Loose-fitting canvas-type truck covers will be prohibited. If loads contain wet material capable of producing free liquid, truck liners will be used.

Before excavation of potentially contaminated soil, a plan will be prepared to include the truck transport route. Trucks loaded with site materials will exit the Site using approved truck routes. This will be the most appropriate route and takes into account: (a) limiting transport through residential areas and past sensitive sites; (b) use of town-mapped truck routes; (c) prohibiting off-site queuing of trucks entering the facility; (d) limiting total distance to major highways; (e) promoting safety in access to highways; (f) overall safety in transport; and (g) community input where necessary.

Trucks will be prohibited from stopping and idling in the residential neighborhood outside the Site. Egress points for truck and equipment transport from the Site will be kept clean of dirt and other materials during site remediation and development. Trucks will queue on site to minimize off-site disturbance. Off-site queuing will be prohibited.

Deleterious material excavated and removed from the Site (if encountered) will be treated as contaminated and regulated material and will be transported and disposed of off-site in a permitted facility in accordance with local, state, and federal regulations. If disposal of such material from the Site is proposed for unregulated off-site disposal (i.e., clean soil removed for development purposes), a formal request with an associated plan will be

made to the NYSDEC project manager. Unregulated off-site management of such materials from the Site will not occur without formal NYSDEC project manager approval.

Off-site disposal locations for excavated soils will be identified in the pre-excavation notification. This will include estimated quantities and a breakdown by class of disposal facility if appropriate (e.g., hazardous waste disposal facility, solid waste landfill, petroleum treatment facility, C&D debris recovery facility). Actual disposal quantities and associated documentation will be reported to the NYSDEC. This documentation will include, but will not be limited to: waste profiles, test results, facility acceptance letters, manifests, bills of lading, and facility receipts.

Non-hazardous historical fill and contaminated soils taken off site will be handled consistent with 6 NYCRR Parts 360 (General requirements for solid waste management facilities), 361(Rules for material recovery facilities (recycling operations), 362 (Rules for combustion, thermal treatment, transfer, and collection facilities), 363 (Regulations for landfills), 364(Requirements for waste transporters) and 365 (Standards for handling regulated medical and other infectious wastes). Material that does not meet Unrestricted Soil Cleanup Objectives (SCOs) is prohibited from being taken to a New York State C&D debris recovery facility (6 NYCRR Subpart 360-15 registered or permitted facility).

4.6 Fluids Management from Excavations

Liquids to be removed from the Site, including but not limited to, excavation dewatering and decontamination waters, will be handled, transported, and disposed offsite at a permitted facility in accordance with applicable local, state, and federal regulations. Dewatering fluids will not be recharged back to the land surface or subsurface of the Site and will be managed off site unless previous approval is obtained from NYSDEC.

Discharge of water generated during large-scale construction to surface waters (e.g., a local pond, stream, or river) will be performed under a State Pollutant Discharge Elimination System (SPDES) permit.

4.7 Cover Systems Restoration

There is currently no cover system on site. If a cover is installed, this section will be updated.

4.8 Stormwater Pollution Prevention

A Stormwater Pollution Prevention Plan for construction activities is included in the Best Management Practice Plan, dated January 20, 2023 **(Appendix E)**.

4.9 Excavation Contingency Plan

If underground tanks or other previously unidentified contaminant sources are found during subsurface excavations or development-related construction, excavation will be suspended until sufficient equipment is mobilized to address the condition. The NYSDEC project manager will be promptly notified of the discovery.

Sampling will be performed on product, sediment, and surrounding soils as necessary to determine the nature of the material and proper disposal method following the Field Sampling Plan **(Appendix B)**. Sampling and associated chemical analysis will be proposed in a separate plan to the NYSDEC for approval before sampling. Tanks will be closed as per NYSDEC regulations and guidance.

Unknown or unexpected contaminated media identified by screening during invasive site work will be promptly communicated by phone within 2 hours to NYSDEC's project manager. Reportable quantities of petroleum product will also be reported to the NYSDEC spills hotline. These findings will be documented in a Construction Completion Report that will be provided to the NYSDEC at the completion of the project. (see Section 4.13).

4.10 Community Air Monitoring Plan

During soil disturbance, the CAMP will be followed. Air sampling stations will be based on generally prevailing wind conditions and areas where work is being conducted. These locations will be adjusted daily or more frequently based on actual wind directions to provide an upwind and at least two downwind air monitoring stations. If a sensitive receptor, such as a school, day care or residential area is adjacent to the Site, a fixed monitoring station should be located at that site perimeter, regardless of wind direction.

Exceedances of action levels listed in the CAMP will be reported to NYSDEC and NYSDOH project managers within 24 hours. The CAMP is provided in **Appendix F**.

4.11 Odor Control Plan

This odor control plan can control emissions of on-site and off-site nuisance odors , and the plan will be updated as necessary following completion of the remedial investigation. Specific odor control methods to be used routinely will be added to this odor control plan following completion of the Remedial Investigation or as needed if odors are identified in the interim. If nuisance odors are identified at the Site boundary, or if odor complaints are received, work will be halted, and the source of odors will be identified and corrected. Work will not resume until nuisance odors have been abated. NYSDEC and NYSDOH will be notified of odor events and of any other complaints about the project. Implementation of odor controls, including the halting of work, is the responsibility of the remedial party's Remediation Engineer, and any measures implemented will be discussed in the Construction Completion Report.

All reasonable means will be employed to prevent on- and off-site nuisances. At a minimum, these measures will include: (a) limiting the area of open excavations and sizes of soil stockpiles and (b) shrouding open excavations with tarps and other covers. If odors develop and cannot be otherwise controlled, additional means to eliminate odor nuisances will include: (a) direct load-out of soils to trucks for off-site disposal; (b) use of chemical odorants in spray or misting systems; and (c) use of staff to monitor odors in surrounding neighborhoods.

If nuisance odors develop during intrusive work that cannot be corrected, or where the control of nuisance odors cannot otherwise be achieved due to on-site conditions or close proximity to sensitive receptors, odor control will be achieved by sheltering the excavation and handling areas in a temporary containment structure equipped with appropriate air venting/filtering systems.

4.12 Dust Control Plan

Particulate monitoring must be conducted according to the CAMP. If particulate levels at the Site exceed the thresholds listed in the CAMP **(Appendix F)**, or if airborne dust is observed on the Site or leaving the Site, the dust suppression techniques listed below will be employed. The construction contractor will also take measures listed below to prevent dust production on the Site.

A dust suppression plan that addresses dust management during invasive on-site work will include, at a minimum, the items listed below:

- Dust suppression will be achieved using a dedicated on-site water truck for road wetting. The truck will be equipped with a water cannon capable of spraying water directly onto off-road areas including excavations and stockpiles.
- Larger sites will be cleared and grubbed in stages to limit the area of exposed, unvegetated soils vulnerable to dust production.
- Gravel will be used on roadways to provide a clean and dust-free road surface.
- On-site roads will be limited in total area to minimize the area required for water truck sprinkling.

The following additional measures could be taken to prevent dust production and suppress dust:

- Covering excavated areas with biodegradable fabric that also can control erosion and serve as a substrate for favorable ecosystems or with synthetic material that can be reused for other on-site or off-site purposes;
- Spraying water in vulnerable areas in conjunction with water conservation and runoff management techniques;
- Securing and covering material in open trucks while hauling excavated material and reusing the covers;
- Revegetating excavated areas as quickly as possible; and

Limiting on-site vehicle speeds.

4.13 Reporting

On submission of the COU, NYSDEC will provide direction on whether reporting is required as described in this section. For projects that require reporting, the following plan will be followed:

- Weekly Field Reports will be required for projects that require soil disturbance and are not considered remediation related. Weekly field Reports will briefly summarize field activities, any soil exports/imports/reuse information, samples collected, problems encountered, complaints received, and CAMP data. These reports will be submitted to NYSDEC and NYSDOH.
- In addition to Weekly Field Reports, Final Construction Completion Reports (CCRs) will be required for projects that require soil disturbance and are considered remediation related within or near AOCs or other potentially contaminated areas (e.g. soil excavation and offsite disposal). The CCRs will include a project summary including descriptions of site excavations/material placement, incident reports, description of CAMP data, waste characterization results, bills of lading/waste manifests, import requests, figures and analytical results. The CCR will be completed in accordance with DER-10.

CCRs and Weekly Field Reports will not be required for importing approved backfill or projects not requiring soil disturbance.

5 References

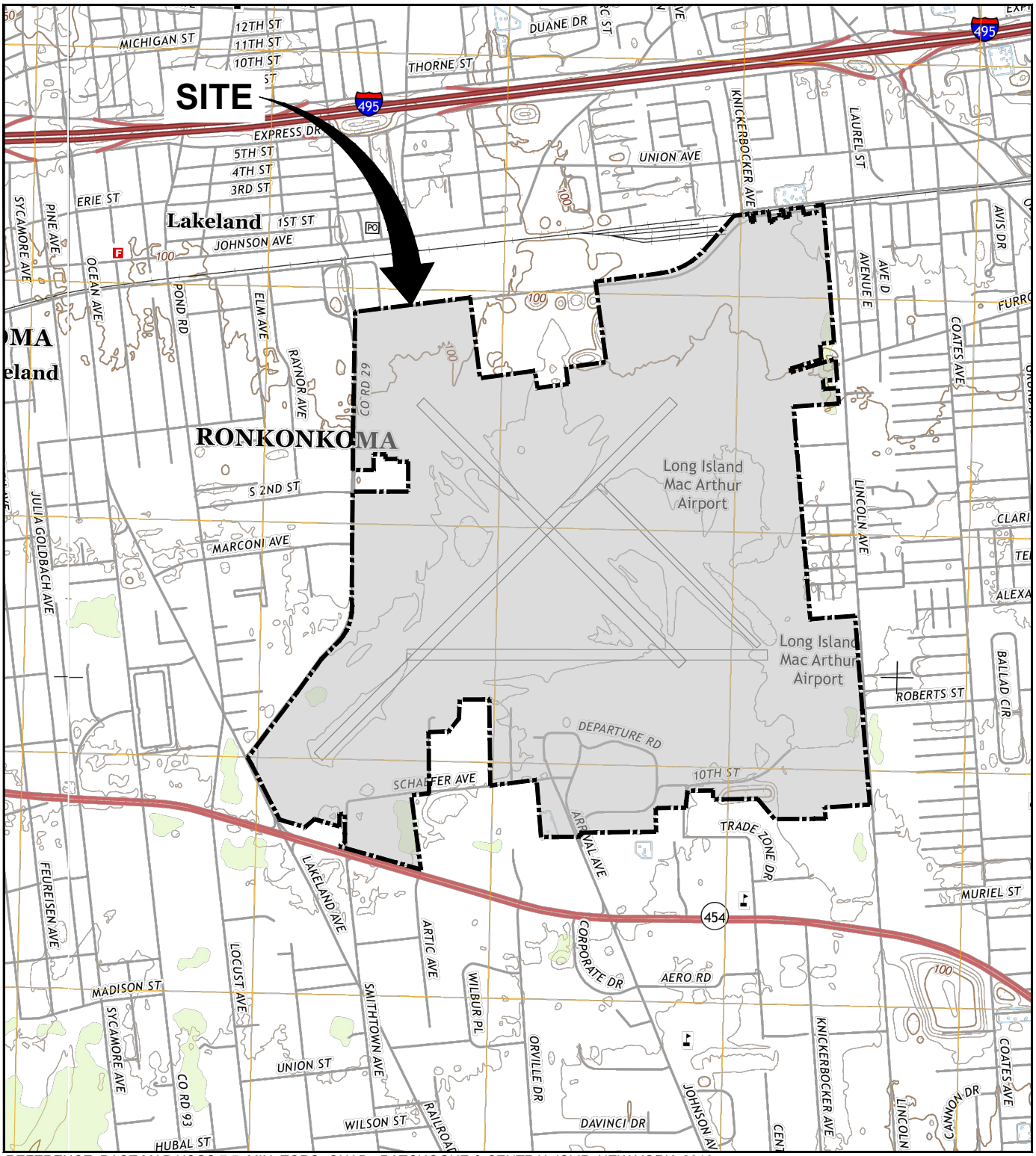
Arcadis 2022. Site Characterization Report, December 2022. Long Island MacArthur Airport. 100 Arrival Ave, Ronkonkoma NY. NYSDEC Site No. 152251.NYSDEC. 2023. Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances. April

Arcadis 2023. Best Management Plan, January 20, 2023. Long Island MacArthur Airport. 100 Arrival Ave, Ronkonkoma NY. NYSDEC Site No. 152251.NYSDEC

NYSDEC's. 2010. Technical Guidance for Site Investigation and Remediation (DER-10). May 3, 2010

NYSDEC. 2011. Green Remediation (DER-31), January 20, 2011.

Figures



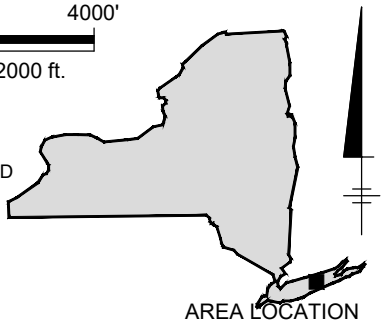
REFERENCE: BASE MAP USGS 7.5. MIN. TOPO. QUAD., PATCHOGUE & CENTRAL ISLIP, NEW YORK, 2016.

0 2000' 4000'

Approximate Scale: 1 in. = 2000 ft.

NOTE:

PARCEL BOUNDARY DATA OBTAINED FROM SUFFOLK COUNTY, REAL PROPERTY TAX SERVICE AGENCY GEOGRAPHICAL INFORMATION SYSTEMS.



AREA LOCATION
NEW YORK

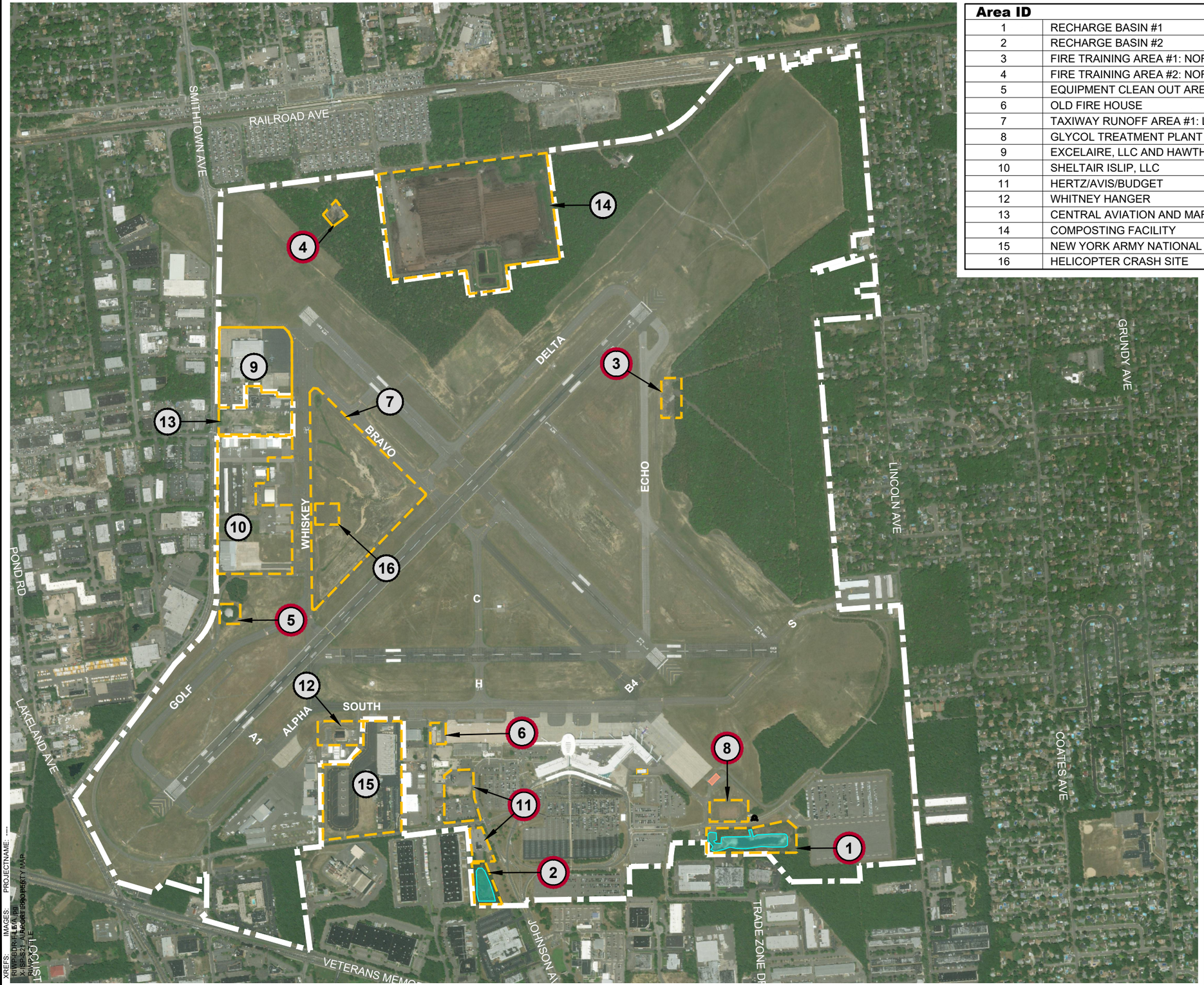
LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NEW YORK
REMEDIAL INVESTIGATION WORK PLAN

SITE LOCATION

ARCADIS

FIGURE
1

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Area ID	
1	RECHARGE BASIN #1
2	RECHARGE BASIN #2
3	FIRE TRAINING AREA #1: NORTHEAST WOODED AREA
4	FIRE TRAINING AREA #2: NORTHWEST, BEHIND COMPOST FACILITY
5	EQUIPMENT CLEAN OUT AREA: BY SUFFOLK COUNTY WATER TOWER
6	OLD FIRE HOUSE
7	TAXIWAY RUNOFF AREA #1: LOW LYING AREA BETWEEN TAXIWAYS WHISKEY AND BRAVO
8	GLYCOL TREATMENT PLANT
9	EXCELAIRE, LLC AND HAWTHORNE GLOBAL AVIATON SERVICES
10	SHELTAIR ISLIP, LLC
11	HERTZ/AVIS/BUDGET
12	WHITNEY HANGER
13	CENTRAL AVIATION AND MARINE CORPORATION (CAMCO)
14	COMPOSTING FACILITY
15	NEW YORK ARMY NATIONAL GUARD AVIATION UNIT
16	HELICOPTER CRASH SITE

LEGEND:

- SITE BOUNDARY
- RECHARGE BASIN
- APPROXIMATE LIMITS OF AREA
- HYDRANT
- (#) ON SITE AREA ID
- AREA RETAINED FOR RI

NOTES:

1. AERIAL PHOTOGRAPHY: © 2024 MICROSOFT CORPORATION © 2024 MAXAR ©CNES (2024) DISTRIBUTION AIRBUS DS
2. PARCEL BOUNDARY DATA OBTAINED FROM SUFFOLK COUNTY, REAL PROPERTY TAX SERVICE AGENCY GEOGRAPHICAL INFORMATION SYSTEMS.
3. REFER TO RECORDS SEARCH REPORT (AUGUST 8TH, 2019) FOR DETAILS ON AREAS AND EXISTING MONITORING WELLS.

0 1000' 2000'

APPROXIMATE SCALE IN FEET

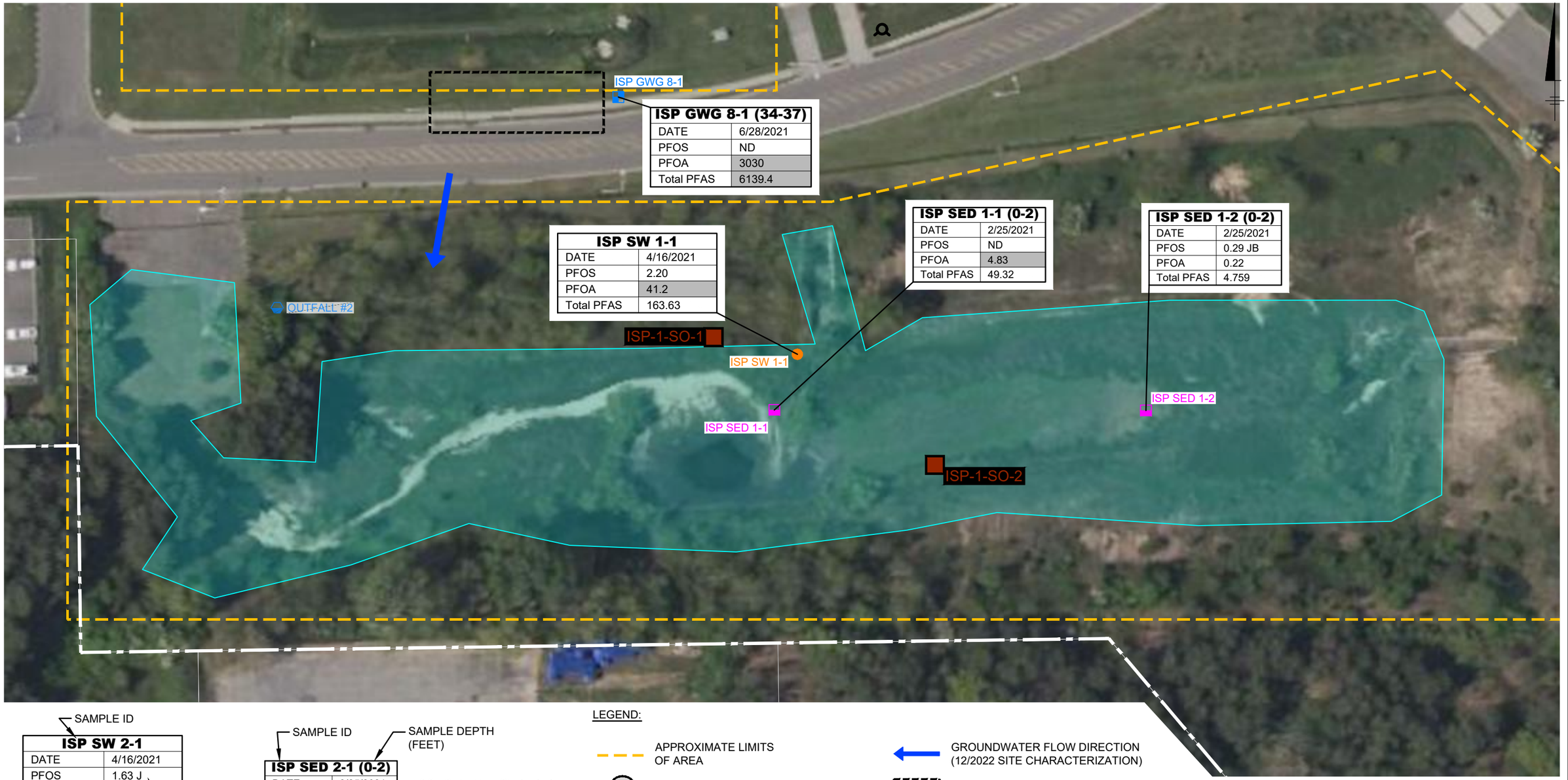
LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NEW YORK
REMEDIAL INVESTIGATION WORK PLAN

SITE LAYOUT AND AREAS

ARCADIS

FIGURE 2

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XREFS: IMAGES: PROJECTNAME: ----
RIWP-BDR-FIG-A.jpg
X-ISP-21_AIRPORT PROPERTY MAP
RIWP-2.DWG
ACTIVE



SAMPLE ID

ISP SW 2-1	
DATE	4/16/2021
PFOS	1.63 J
PFOA	1.45 J
Total PFAS	33.42

CONSTITUENT

CONCENTRATION (ng/L), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

SAMPLE ID

SAMPLE DEPTH (FEET)

ISP SED 2-1 (0-2)	
DATE	2/25/2021
PFOS	6.13
PFOA	0.95 J
Total PFAS	55.44

CONCENTRATION (µg/kg), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

CONSTITUENT

NYS PFAS Guidance Values				
	Commercial (µg/kg)	Industrial (µg/kg)	Protection of Groundwater (µg/kg)	Ambient Water Quality (ng/L)
PFOA	500	600	0.8	6.7
PFOS	440	440	1.0	2.7

LEGEND:

- APPROXIMATE LIMITS OF AREA
- (#) ON SITE AREA ID
- APPROXIMATE OUTFALL LOCATION
- SEDIMENT SAMPLE LOCATION
- STORMWATER SAMPLE LOCATION
- GROUNDWATER GRAB SAMPLE LOCATION
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS)
- GROUNDWATER FLOW DIRECTION (12/2022 SITE CHARACTERIZATION)
- AREA CONTAINING UTILITIES
- RECHARGE BASIN

0 60' 120'

APPROXIMATE SCALE IN FEET

NOTES:

1. GROUNDWATER AND SURFACE WATER SAMPLES ARE IN ng/L
2. SOIL SAMPLES ARE REPORTED IN µg/Kg
3. PROPOSED SAMPLE LOCATIONS ARE APPROXIMATE AND WILL BE SUBJECT TO CHANGE BASED ON SITE CONDITIONS.
4. SAMPLE LOCATIONS WILL BE SURVEYED FOLLOWING COMPLETION OF SAMPLING EVENT

LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NEW YORK
REMEDIAL INVESTIGATION WORK PLAN

AREA 1: RECHARGE BASIN #1 AND AREA 8 GLYCOL TREATMENT PLANT

ARCADIS

FIGURE 3

C:\Users\mwallewski\DCACCDocs\Arcadis ACC USAUS-99999999-TOWN OF ISLIP_MACARTHUR AIRPORT_ISLIP_NY\Project Files\10_WIP\10T_ARC_ENV\2025\01-DWG\RIWP-F04 - AREA 01.dwg LAYOUT: 4 SAVED: 5/14/2025 8:24 AM ACADVER: 24.2S (LMS TECH) PAGESETUP: ---
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X-BDR-F-LE LIMA.jpg
X-ISP-S21_AIRPORT PROPERTY MAP
RIWP-X-TITLE



ISP SW 2-1	
DATE	4/16/2021
PFOS	1.63 J
PFOA	1.45 J
Total PFAS	33.42

ISP SED 2-1 (0-2)	
DATE	2/25/2021
PFOS	6.13
PFOA	0.95 J
Total PFAS	55.44

ISP SED 2-2 (0-2)	
DATE	2/25/2021
PFOS	0.47 J
PFOA	ND
Total PFAS	2.897

LEGEND:

- SITE BOUNDARY
- APPROXIMATE LIMITS OF AREA
- (#) ON SITE AREA ID
- SEDIMENT SAMPLE LOCATION
- STORMWATER SAMPLE LOCATION
- GROUNDWATER GRAB SAMPLE LOCATION
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLE ABOVE WATER TABLE
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLE ABOVE WATER TABLE. CO-LOCATED WITH A GROUNDWATER GRAB SAMPLE
- PROPOSED VAP LOCATION
- GROUNDWATER FLOW DIRECTION (12/2022 SITE CHARACTERIZATION)
- RECHARGE BASIN

SAMPLE ID

ISP SW 2-1	
DATE	4/16/2021
PFOS	1.63 J
PFOA	1.45 J
Total PFAS	33.42

CONSTITUENT

CONCENTRATION (ng/L), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

SAMPLE ID

SAMPLE DEPTH (FEET)

ISP SED 2-1 (0-2)	
DATE	2/25/2021
PFOS	6.13
PFOA	0.95 J
Total PFAS	55.44

CONSTITUENT

CONCENTRATION (µg/kg), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

NYS PFAS Guidance Values				
	Commercial (µg/kg)	Industrial (µg/kg)	Protection of Groundwater (µg/kg)	Ambient Water Quality (ng/L)
PFOA	500	600	0.8	6.7
PFOS	440	440	1.0	2.7

NOTES:

1. GROUNDWATER AND SURFACE WATER SAMPLES ARE REPORTED IN ng/L
2. SOIL SAMPLES ARE REPORTED IN µg/kg
3. PROPOSED SAMPLE LOCATIONS ARE APPROXIMATE AND WILL BE SUBJECT TO CHANGE BASED ON SITE CONDITIONS.
4. SAMPLE LOCATIONS WILL BE SURVEYED FOLLOWING COMPLETION OF SAMPLING EVENT

0 50' 100'

APPROXIMATE SCALE IN FEET

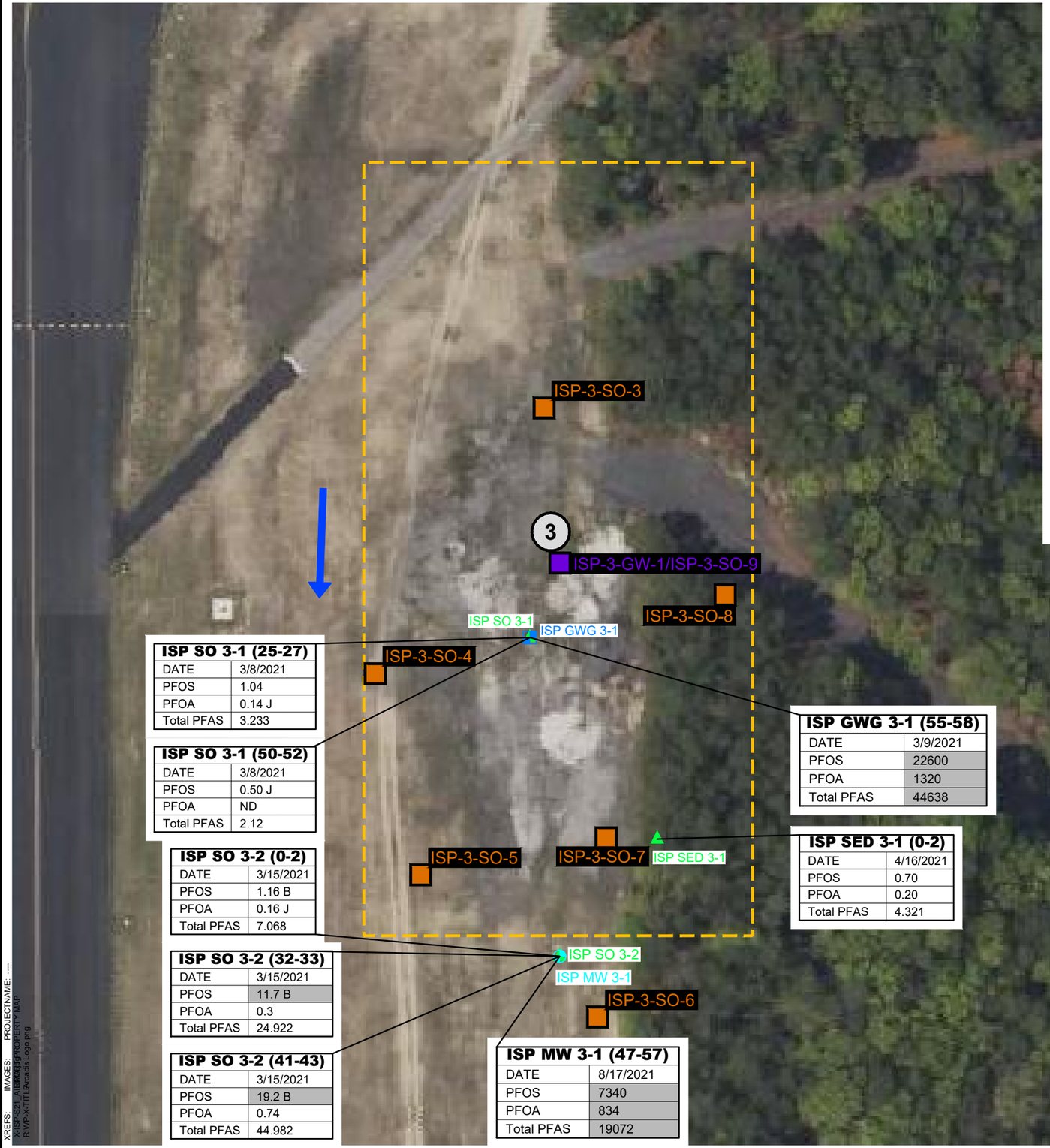
LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NEW YORK
REMEDIAL INVESTIGATION WORK PLAN

AREA 2:
RECHARGE BASIN #2

ARCADIS

FIGURE
4

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XREFS: IMAGES: PROJECTNAME: ----
XI\ISP-S21_AERIAL.jpg XI\PROPERTY MAP RIWP-X-TITLE\Arcadis Logo.png



ISP SO 3-1 (25-27)	
DATE	3/8/2021
PFOS	1.04
PFOA	0.14 J
Total PFAS	3.233

ISP SO 3-1 (50-52)	
DATE	3/8/2021
PFOS	0.50 J
PFOA	ND
Total PFAS	2.12

ISP SO 3-2 (0-2)	
DATE	3/15/2021
PFOS	1.16 B
PFOA	0.16 J
Total PFAS	7.068

ISP SO 3-2 (32-33)	
DATE	3/15/2021
PFOS	11.7 B
PFOA	0.3
Total PFAS	24.922

ISP SO 3-2 (41-43)	
DATE	3/15/2021
PFOS	19.2 B
PFOA	0.74
Total PFAS	44.982

ISP GWG 3-1 (55-58)	
DATE	3/9/2021
PFOS	22600
PFOA	1320
Total PFAS	44638

ISP SED 3-1 (0-2)	
DATE	4/16/2021
PFOS	0.70
PFOA	0.20
Total PFAS	4.321

ISP MW 3-1 (47-57)	
DATE	8/17/2021
PFOS	7340
PFOA	834
Total PFAS	19072

LEGEND:

- SITE BOUNDARY
- APPROXIMATE LIMITS OF AREA
- (#) ON SITE AREA ID
- SOIL BORING LOCATION
- MONITORING WELL LOCATION
- GROUNDWATER GRAB SAMPLE LOCATION
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLE ABOVE WATER TABLE
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLES EVERY 10 FT TO THE WATER TABLE. CO-LOCATED WITH VAP LOCATION.
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLES EVERY 10 FT TO THE WATER TABLE
- GROUNDWATER FLOW DIRECTION

SAMPLE ID

ISP SO 3-1 (50-52)	
DATE	3/8/2021
PFOS	0.50 J
PFOA	ND
Total PFAS	2.12

CONCENTRATION (ng/L), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

CONSTITUENT

SAMPLE ID, SAMPLE DEPTH (FEET)

ISP SED 3-1 (0-2)	
DATE	3/8/2021
PFOS	0.7
PFOA	0.2
Total PFAS	4.321

CONCENTRATION (µg/kg), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

CONSTITUENT

NYS PFAS Guidance Values				
	Commercial (µg/kg)	Industrial (µg/kg)	Protection of Groundwater (µg/kg)	Ambient Water Quality (ng/L)
PFOA	500	600	0.8	6.7
PFOS	440	440	1.0	2.7

NOTES:

1. REPORTED GROUNDWATER AND SURFACE WATER SAMPLES ARE IN ng/L
2. SOIL SAMPLES ARE REPORTED IN µg/Kg
3. GROUNDWATER AND SURFACE WATER SAMPLES ARE REPORTED IN ng/L
4. SOIL SAMPLES ARE REPORTED IN µg/Kg
5. PROPOSED SAMPLE LOCATIONS ARE APPROXIMATE AND WILL BE SUBJECT TO CHANGE BASED ON SITE CONDITIONS.
6. SAMPLE LOCATIONS WILL BE SURVEYED FOLLOWING COMPLETION OF SAMPLING EVENT
7. UP TO 5 PROVISIONAL STEP OUT LOCATIONS PROPOSED FOR THIS AOC AND ARE NOT SHOWN IN THIS FIGURE. LOCATIONS OF PROVISIONAL SAMPLES, IF NEEDED, WILL BE BASED ON RESULTS FROM PRIMARY SOIL SAMPLES.

0 60' 120'

APPROXIMATE SCALE IN FEET

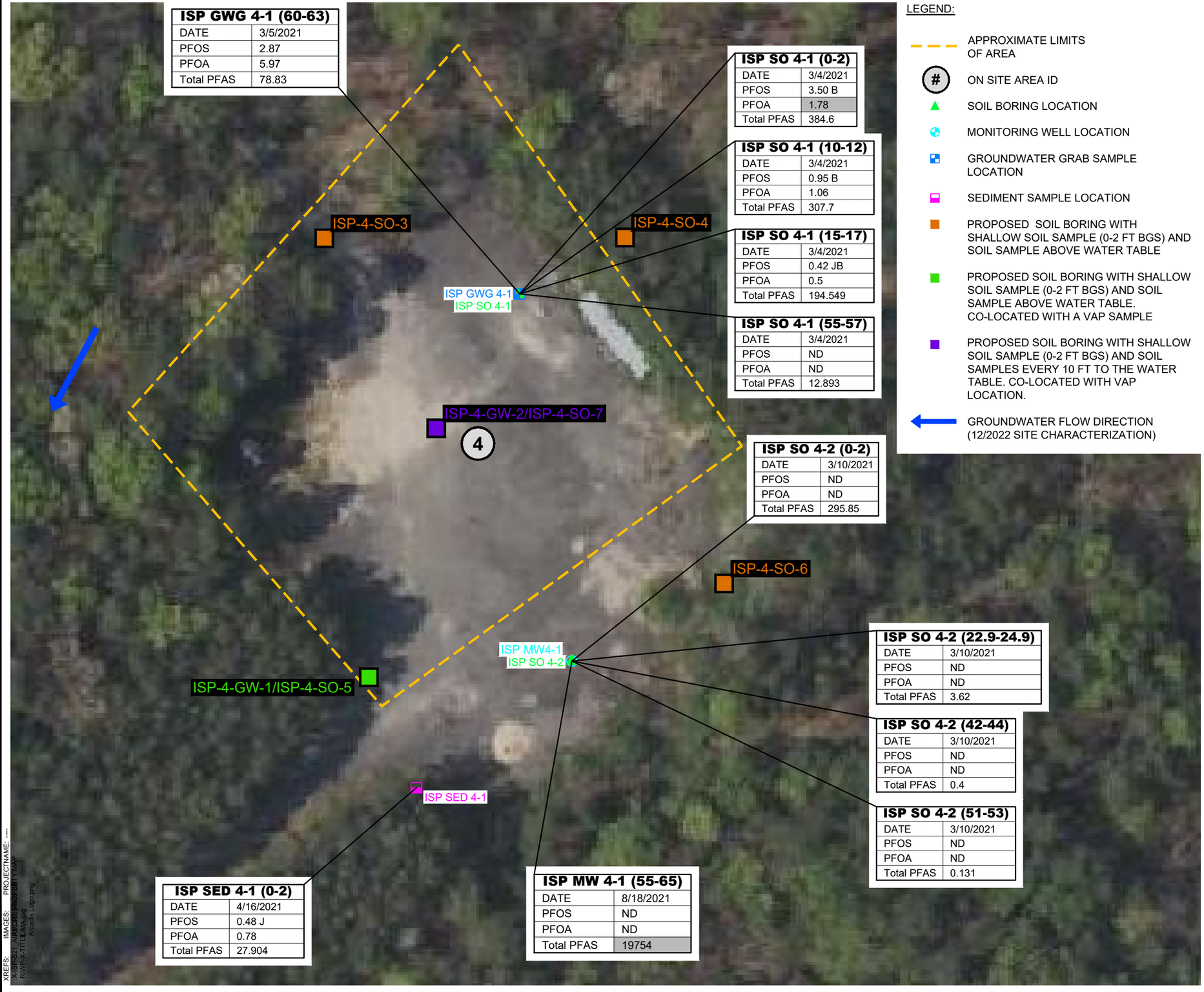
LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NEW YORK
REMEDIAL INVESTIGATION WORK PLAN

AREA 3:
FIRE TRAINING AREA #1

ARCADIS

FIGURE
5

CITY:(Reqd) DN/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
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XREFS: IMAGES: PROJECTNAME: ----
X-ISP-S21 AIRPORTPROPERTY MAP
RIWP-X-TITLArcadis Logo.png



ISP GWG 4-1 (60-63)	
DATE	3/5/2021
PFOS	2.87
PFOA	5.97
Total PFAS	78.83

ISP SO 4-1 (0-2)	
DATE	3/4/2021
PFOS	3.50 B
PFOA	1.78
Total PFAS	384.6

ISP SO 4-1 (10-12)	
DATE	3/4/2021
PFOS	0.95 B
PFOA	1.06
Total PFAS	307.7

ISP SO 4-1 (15-17)	
DATE	3/4/2021
PFOS	0.42 JB
PFOA	0.5
Total PFAS	194.549

ISP SO 4-1 (55-57)	
DATE	3/4/2021
PFOS	ND
PFOA	ND
Total PFAS	12.893

ISP SO 4-2 (0-2)	
DATE	3/10/2021
PFOS	ND
PFOA	ND
Total PFAS	295.85

ISP SO 4-2 (22.9-24.9)	
DATE	3/10/2021
PFOS	ND
PFOA	ND
Total PFAS	3.62

ISP SO 4-2 (42-44)	
DATE	3/10/2021
PFOS	ND
PFOA	ND
Total PFAS	0.4

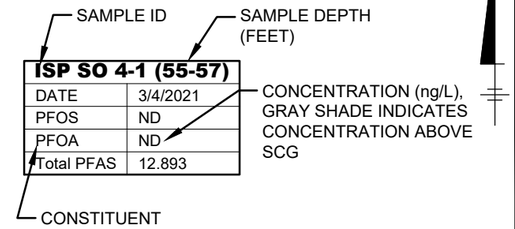
ISP SO 4-2 (51-53)	
DATE	3/10/2021
PFOS	ND
PFOA	ND
Total PFAS	0.131

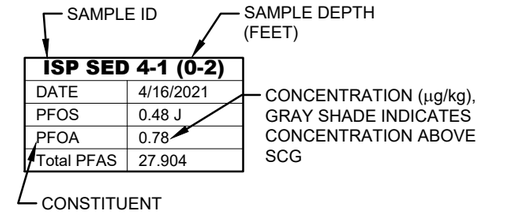
ISP SED 4-1 (0-2)	
DATE	4/16/2021
PFOS	0.48 J
PFOA	0.78
Total PFAS	27.904

ISP MW 4-1 (55-65)	
DATE	8/18/2021
PFOS	ND
PFOA	ND
Total PFAS	19754

LEGEND:

- APPROXIMATE LIMITS OF AREA
- (#) ON SITE AREA ID
- SOIL BORING LOCATION
- MONITORING WELL LOCATION
- GROUNDWATER GRAB SAMPLE LOCATION
- SEDIMENT SAMPLE LOCATION
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLE ABOVE WATER TABLE
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLE ABOVE WATER TABLE. CO-LOCATED WITH A VAP SAMPLE
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLES EVERY 10 FT TO THE WATER TABLE. CO-LOCATED WITH VAP LOCATION.
- GROUNDWATER FLOW DIRECTION (12/2022 SITE CHARACTERIZATION)





NYS PFAS Guidance Values				
	Commercial (µg/kg)	Industrial (µg/kg)	Protection of Groundwater (µg/kg)	Ambient Water Quality (ng/L)
PFOA	500	600	0.8	6.7
PFOS	440	440	1.0	2.7

NOTES:

1. GROUNDWATER AND SW SAMPLES ARE REPORTED IN ng/L
2. SOIL SAMPLES ARE REPORTED IN µg/kg
3. PROPOSED SAMPLE LOCATIONS ARE APPROXIMATE AND WILL BE SUBJECT TO CHANGE BASED ON SITE CONDITIONS.
4. SAMPLE LOCATIONS WILL BE SURVEYED FOLLOWING COMPLETION OF SAMPLING EVENT
5. UP TO 2 PROVISIONAL STEP OUT LOCATIONS PROPOSED FOR THIS AREA AND ARE NOT SHOWN IN THIS FIGURE. LOCATIONS OF PROVISIONAL SAMPLES, IF NEEDED, WILL BE BASED ON RESULTS FROM PRIMARY SOIL SAMPLES.

0 60' 120'

APPROXIMATE SCALE IN FEET

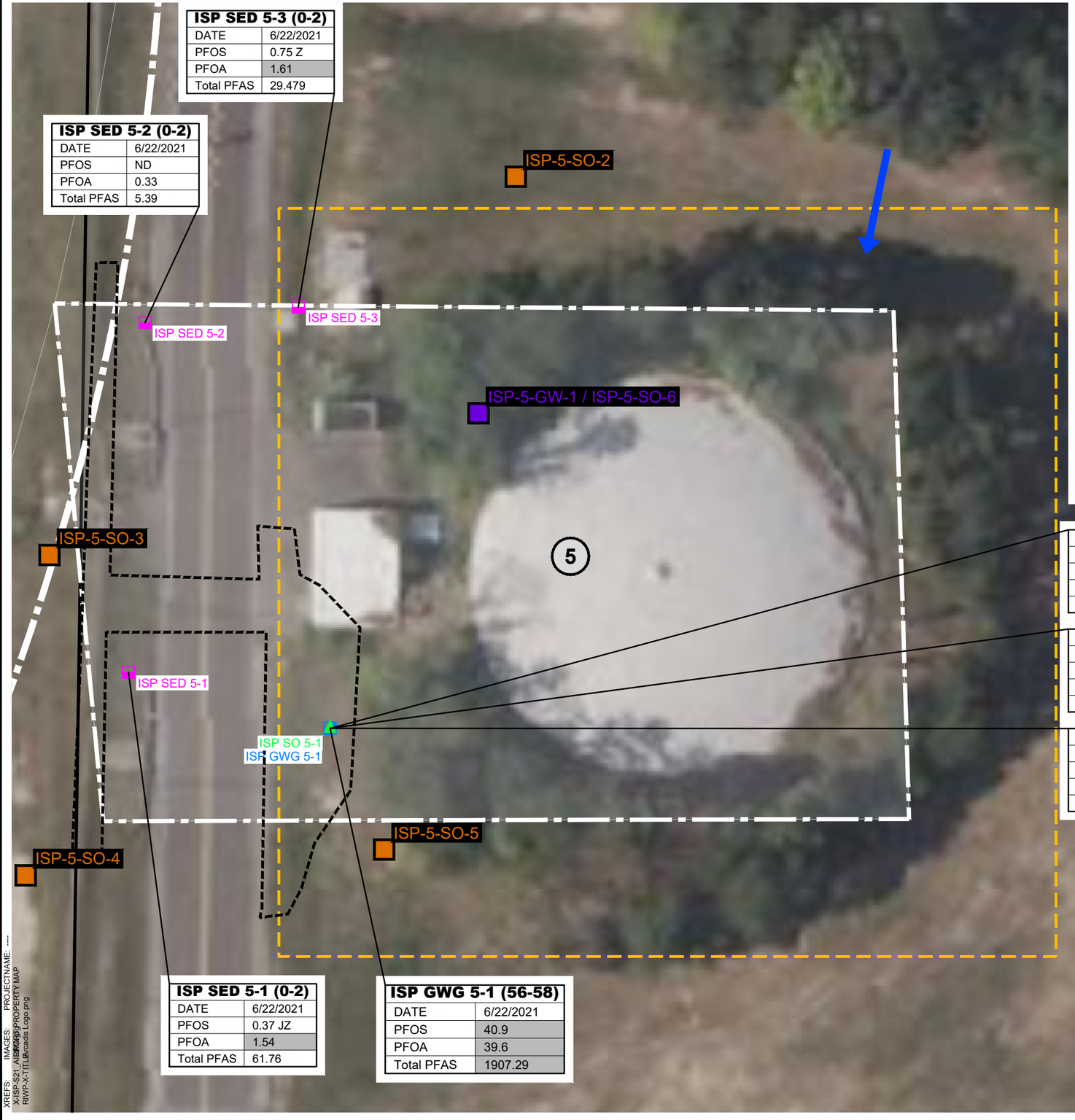
LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NEW YORK
REMEDIAL INVESTIGATION WORK PLAN

AREA 4:
FIRE TRAINING AREA #2

ARCADIS

FIGURE
6

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XREFS: IMAGES: PROJECTNAME: ----
X-ISP-S21 AIRPORT BASE MAP
RIWP-X-TITLE IMA.jpg
Arcadis Logo.png



ISP SED 5-3 (0-2)	
DATE	6/22/2021
PFOS	0.75 Z
PFOA	1.61
Total PFAS	29.479

ISP SED 5-2 (0-2)	
DATE	6/22/2021
PFOS	ND
PFOA	0.33
Total PFAS	5.39

ISP SED 5-1 (0-2)	
DATE	6/22/2021
PFOS	0.37 JZ
PFOA	1.54
Total PFAS	61.76

ISP GWG 5-1 (56-58)	
DATE	6/22/2021
PFOS	40.9
PFOA	39.6
Total PFAS	1907.29

ISP SO 5-1 (0-2)	
DATE	6/21/2021
PFOS	2.41
PFOA	0.3
Total PFAS	7.777

ISP SO 5-1 (20-22)	
DATE	6/21/2021
PFOS	2.11
PFOA	2.09
Total PFAS	40.606

ISP SO 5-1 (53-55)	
DATE	6/21/2021
PFOS	ND
PFOA	1.23
Total PFAS	37.459

LEGEND:

- SITE BOUNDARY
- APPROXIMATE LIMITS OF AREA
- (#) ON SITE AREA ID
- SOIL BORING LOCATION
- GROUNDWATER GRAB SAMPLE LOCATION
- SEDIMENT SAMPLE LOCATION
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLE ABOVE WATER TABLE
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLES EVERY 10 FT TO THE WATER TABLE. CO-LOCATED WITH VAP LOCATION.
- GROUNDWATER FLOW DIRECTION (12/2022 SITE CHARACTERIZATION)
- AREA CONTAINING UTILITIES

SAMPLE ID

ISP GWG 5-1 (56-58)	
DATE	6/22/2021
PFOS	40.9
PFOA	39.6
Total PFAS	1907.29

CONSTITUENT

CONCENTRATION (ng/L), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

SAMPLE ID — SAMPLE DEPTH (FEET)

ISP SED 5-3 (0-2)	
DATE	6/22/2021
PFOS	0.75 Z
PFOA	1.61
Total PFAS	29.479

CONSTITUENT

CONCENTRATION (µg/kg), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

NYS PFAS Guidance Values				
	Commercial (µg/kg)	Industrial (µg/kg)	Protection of Groundwater (µg/kg)	Ambient Water Quality (ng/L)
PFOA	500	600	0.8	6.7
PFOS	440	440	1.0	2.7

NOTES:

1. GROUNDWATER AND SURFACE WATER SAMPLES ARE REPORTED IN ng/L
2. SOIL SAMPLES ARE REPORTED IN µg/Kg
3. PROPOSED SAMPLE LOCATIONS ARE APPROXIMATE AND WILL BE SUBJECT TO CHANGE BASED ON SITE CONDITIONS.
4. SAMPLE LOCATIONS WILL BE SURVEYED FOLLOWING COMPLETION OF SAMPLING EVENT
5. UP TO 2 PROVISIONAL STEP OUT LOCATIONS PROPOSED FOR THIS AREA AND ARE NOT SHOWN IN THIS FIGURE. LOCATIONS OF PROVISIONAL SAMPLES, IF NEEDED, WILL BE BASED ON RESULTS FROM PRIMARY SOIL SAMPLES.

0 30' 60'

APPROXIMATE SCALE IN FEET

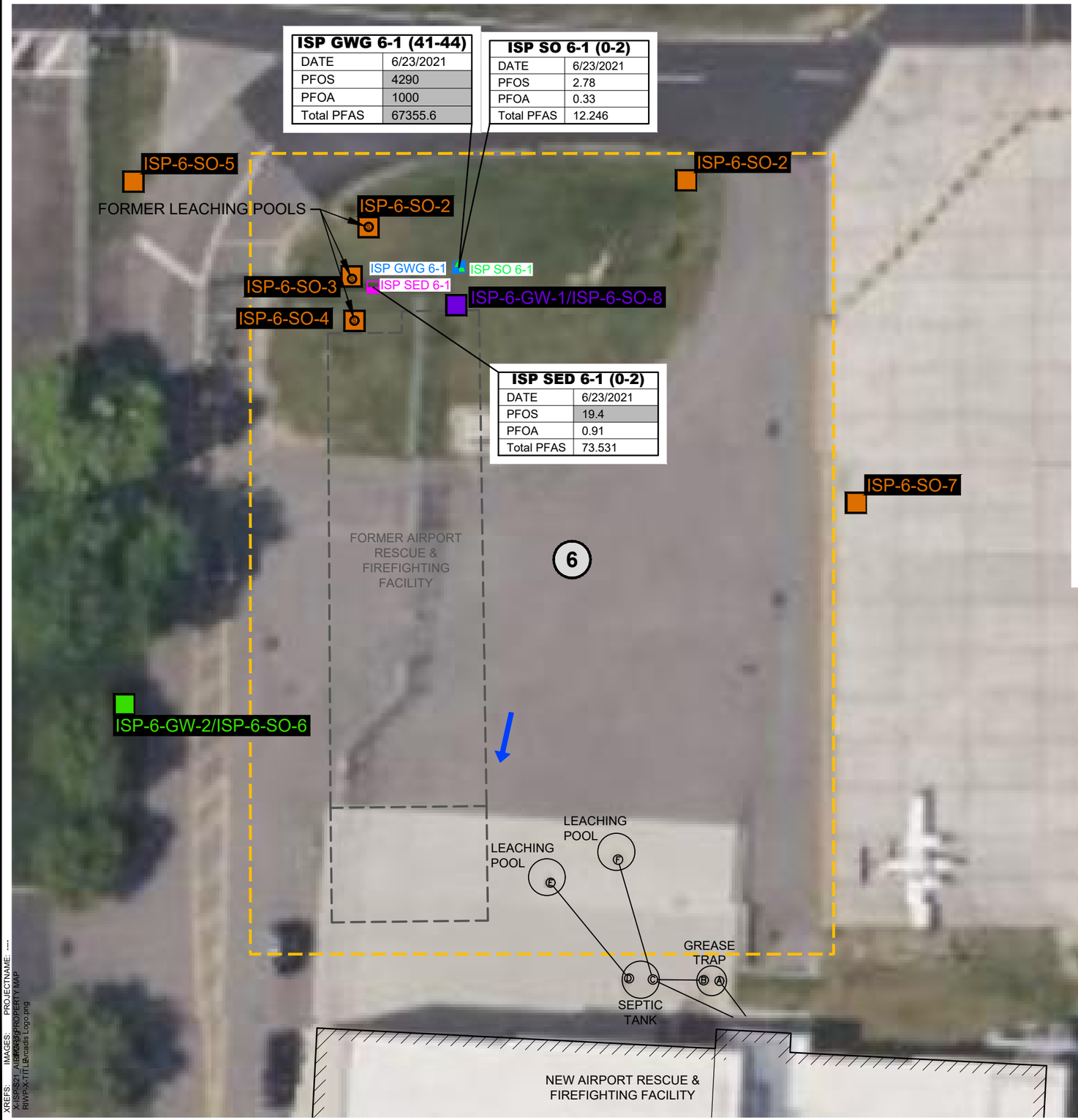
LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NEW YORK
REMEDIAL INVESTIGATION WORK PLAN

AREA 5: EQUIPMENT CLEANOUT AREA BY SUFFOLK COUNTY WATER AUTHORITY

ARCADIS

FIGURE 7

CITY:(Reqd) DIV/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
C:\Users\mwasilewski\DCACCDocs\Arcadis ACC USAUS-99999999-TOWN OF ISLIP_MACARTHUR AIRPORT_ISLIP_NY\Project Files\10_WIP\10T_ARC_ENV\2025\01-DWG\RIWP-F08 - AREA 05.dwg LAYOUT: 8 SAVED: 5/14/2025 8:30 AM ACADVER: 24.2S (LMS TECH) PAGESETUP: ---
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XREFS: IMAGES: PROJECTNAME: ---
X-ISP-S21 AIRPORT PROPERTY MAP
RIWP-X-TITLE Arcadis Logo.png



LEGEND:

- SITE BOUNDARY
- APPROXIMATE LIMITS OF AREA
- (#) ON SITE AREA ID
- SOIL BORING LOCATION
- GROUNDWATER GRAB SAMPLE LOCATION
- SEDIMENT SAMPLE LOCATION
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLE ABOVE WATER TABLE
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLES EVERY 10 FT TO THE WATER TABLE. CO-LOCATED WITH VAP LOCATION.
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLE ABOVE WATER TABLE. CO-LOCATED WITH A VAP SAMPLE
- GROUNDWATER FLOW DIRECTION (12/2022 SITE CHARACTERIZATION)
- AREA CONTAINING UTILITIES

SAMPLE ID

ISP GWG 6-1 (41-44)	
DATE	6/23/2021
PFOS	4290
PFOA	1000
Total PFAS	67355.6

CONSTITUENT

CONCENTRATION (ng/L), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

SAMPLE ID

SAMPLE DEPTH (FEET)

ISP SED 6-1 (0-2)	
DATE	6/23/2021
PFOS	19.4
PFOA	0.91
Total PFAS	73.531

CONCENTRATION (µg/kg), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

CONSTITUENT

NYS PFAS Guidance Values				
	Commercial (µg/kg)	Industrial (µg/kg)	Protection of Groundwater (µg/kg)	Ambient Water Quality (ng/L)
PFOA	500	600	0.8	6.7
PFOS	440	440	1.0	2.7

NOTES:

1. GROUNDWATER AND SURFACE WATER SAMPLES ARE REPORTED IN ng/L
2. SOIL SAMPLES ARE REPORTED IN µg/Kg
3. PROPOSED SAMPLE LOCATIONS ARE APPROXIMATE AND WILL BE SUBJECT TO CHANGE BASED ON SITE CONDITIONS.
4. SAMPLE LOCATIONS WILL BE SURVEYED FOLLOWING COMPLETION OF SAMPLING EVENT

0 30' 60'

APPROXIMATE SCALE IN FEET

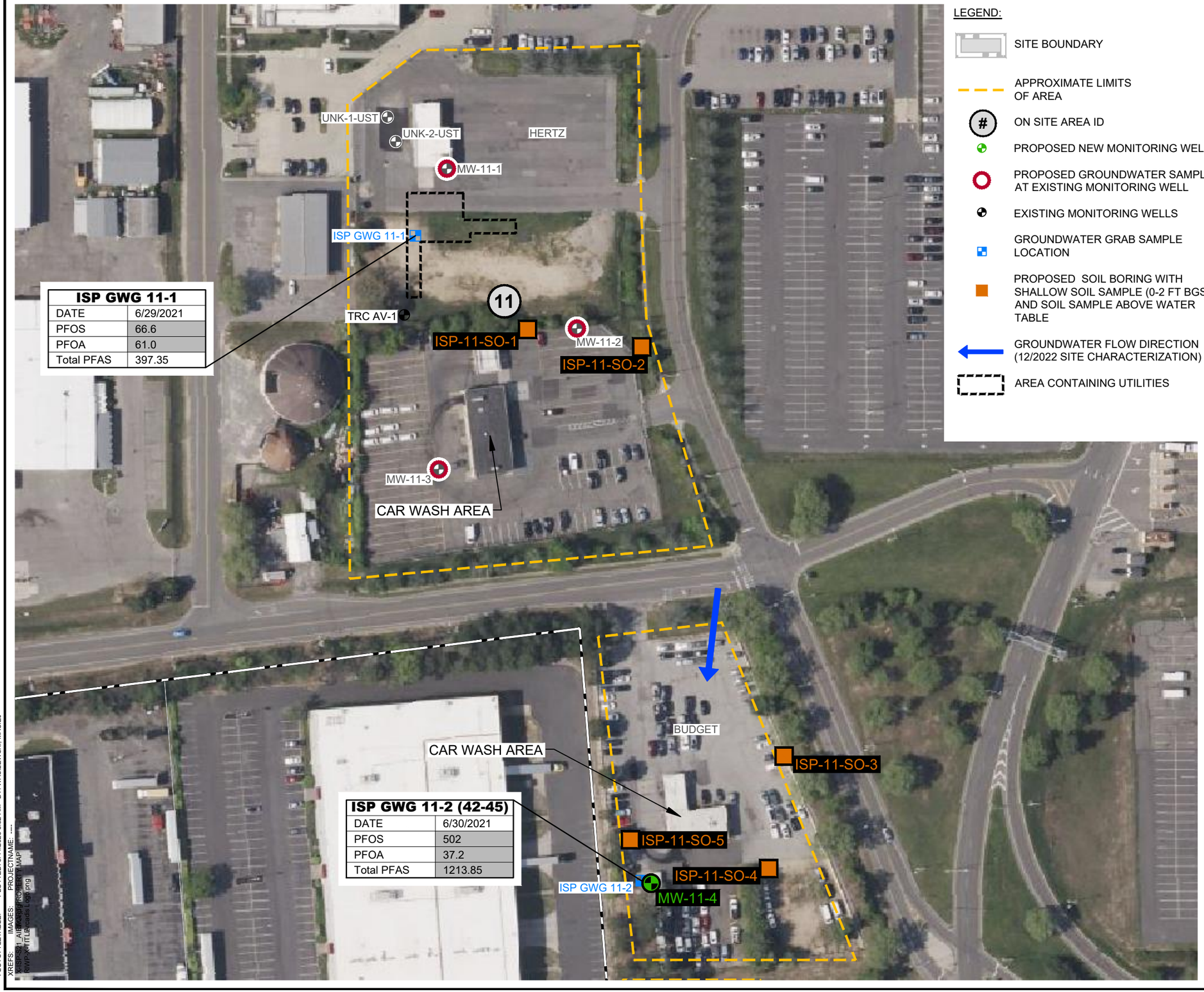
LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NEW YORK
REMEDIAL INVESTIGATION WORK PLAN

AREA 6: OLD FIRE HOUSE & NEW FIRE HOUSE

ARCADIS

FIGURE
8

CITY:(Reqd) DIV/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
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XREFS: IMAGES: PROJECTNAME: ----
X ISP-S21_AIRPORT PROPERTY MAP
R1WPX-TITLArcadis Logo.png



LEGEND:

- SITE BOUNDARY
- APPROXIMATE LIMITS OF AREA
- ON SITE AREA ID
- PROPOSED NEW MONITORING WELLS
- PROPOSED GROUNDWATER SAMPLE AT EXISTING MONITORING WELL
- EXISTING MONITORING WELLS
- GROUNDWATER GRAB SAMPLE LOCATION
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLE ABOVE WATER TABLE
- GROUNDWATER FLOW DIRECTION (12/2022 SITE CHARACTERIZATION)
- AREA CONTAINING UTILITIES

SAMPLE ID

ISP GWG 11-1	
DATE	6/29/2021
PFOS	66.6
PFOA	61.0
Total PFAS	397.35

CONSTITUENT

CONCENTRATION (ng/L), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

SAMPLE ID

SAMPLE DEPTH (FEET)

ISP SED 6-1 (0-2)	
DATE	6/23/2021
PFOS	19.4
PFOA	0.91
Total PFAS	73.531

CONSTITUENT

CONCENTRATION (µg/kg), GRAY SHADE INDICATES CONCENTRATION ABOVE SCG

NYS PFAS Guidance Values				
	Commercial (µg/kg)	Industrial (µg/kg)	Protection of Groundwater (µg/kg)	Ambient Water Quality (ng/L)
PFOA	500	600	0.8	6.7
PFOS	440	440	1.0	2.7

NOTES:

1. GROUNDWATER AND SURFACE WATER SAMPLES ARE REPORTED IN g/L
2. SOIL SAMPLES ARE REPORTED IN µg/Kg
3. PROPOSED SAMPLE LOCATIONS ARE APPROXIMATE AND WILL BE SUBJECT TO CHANGE BASED ON SITE CONDITIONS.
4. SAMPLE LOCATIONS WILL BE SURVEYED FOLLOWING COMPLETION OF SAMPLING EVENT

0 100' 200'

APPROXIMATE SCALE IN FEET

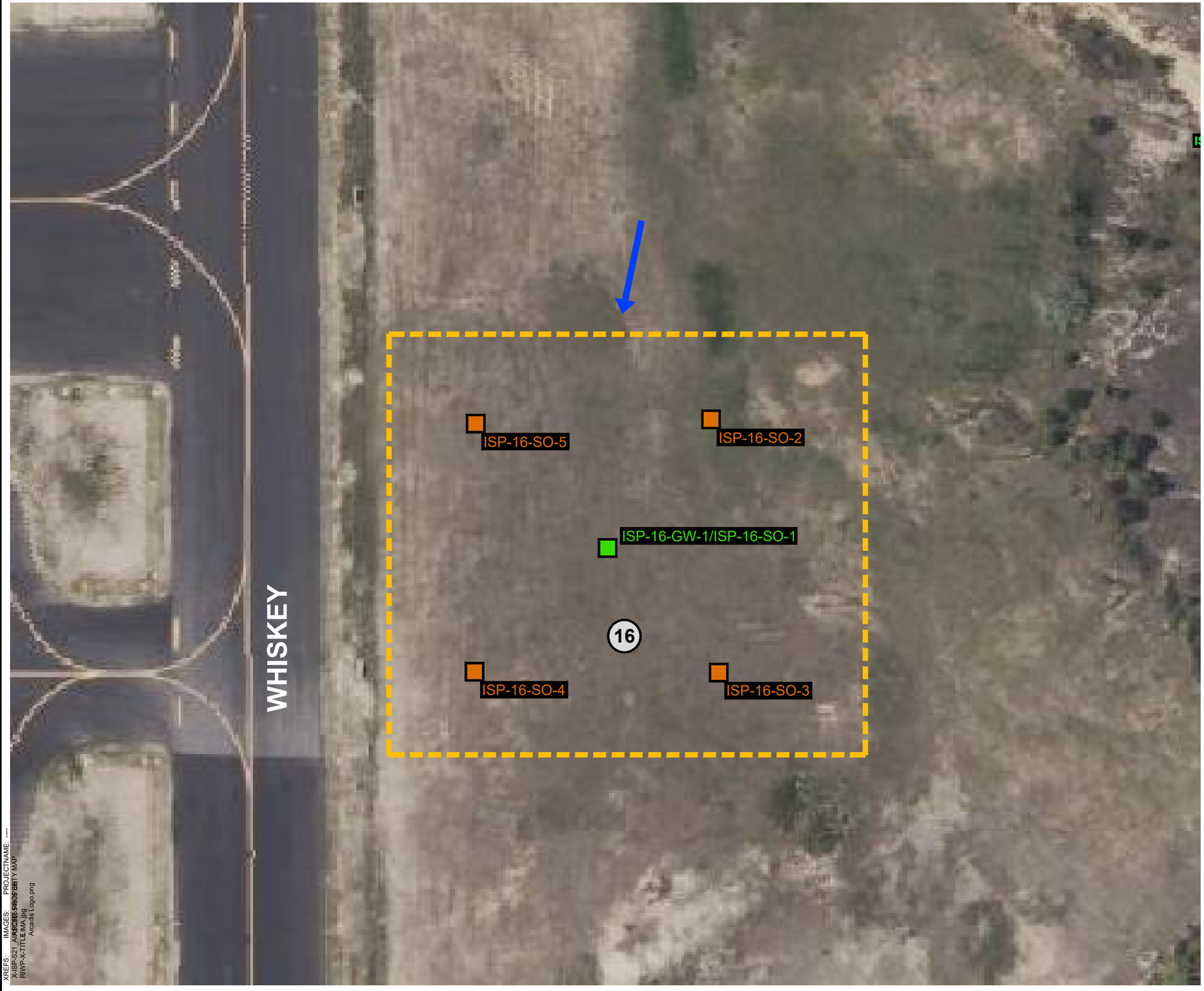
LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NEW YORK
REMEDIAL INVESTIGATION WORK PLAN

AREA 11: HERTZ/AVIS/BUDGET

ARCADIS

FIGURE 9

CITY:(Reqd) DW/GROUP:(Reqd) DB:(Reqd) LD:(Opt) PIC:(Opt) PM:(Reqd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
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PLOTSTYLETABLE: ---- PLOTTED: 5/14/2025 8:32 AM BY: WASILEWSKI, MACIEJ
XREFS: IMAGES: PROJECTNAME: ----
XISIPS-11_AIRBOARD_PROJECT_MAP
R1WP-XTITLE-Arcadis Logo.png



LEGEND:

- APPROXIMATE LIMITS OF AREA
- (#) ON SITE AREA ID
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLE ABOVE WATER TABLE
- PROPOSED SOIL BORING WITH SHALLOW SOIL SAMPLE (0-2 FT BGS) AND SOIL SAMPLE ABOVE WATER TABLE. CO-LOCATED WITH A VAP SAMPLE
- GROUNDWATER FLOW DIRECTION (12/2022 SITE CHARACTERIZATION)

NOTES:

1. PROPOSED SAMPLE LOCATIONS ARE APPROXIMATE AND WILL BE SUBJECT TO CHANGE BASED ON SITE CONDITIONS.
2. SAMPLE LOCATIONS WILL BE SURVEYED FOLLOWING COMPLETION OF SAMPLING EVENT
3. UP TO 2 PROVISIONAL STEP OUT LOCATIONS PROPOSED FOR THIS AREA AND ARE NOT SHOWN IN THIS FIGURE. LOCATIONS OF PROVISIONAL SAMPLES, IF NEEDED, WILL BE BASED ON RESULTS FROM PRIMARY SOIL SAMPLES.

0 50' 100'

APPROXIMATE SCALE IN FEET

LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NEW YORK
REMEDIAL INVESTIGATION WORK PLAN

AREA 16:
CRASH SITE

ARCADIS

FIGURE
10

CITY:(Read) DIV/GROUP:(Read) DB:(Read) LD:(Opt) PIC:(Opt) PM:(Read) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
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XREFS: IMAGES: PROJECTNAME: ----
X-ISP-S21_AIRPORT_SUBPROPERTY MAP
RIWP-X-TITLEIMA.jpg
Arcadis Logo.png

AREAS OF POTENTIAL CONCERN	
AREA ID	**LOCATION DESCRIPTION**
1	RECHARGE BASIN #1
2	RECHARGE BASIN #2
3	FIRE TRAINING AREA #1: NORTHEAST WOODED AREA
4	FIRE TRAINING AREA #2: NORTHWEST, BEHIND COMPOST FACILITY
5	EQUIPMENT CLEAN OUT AREA: BY SUFFOLK COUNTY WATER TOWER
6	OLD FIRE HOUSE
7	TAXIWAY RUNOFF AREA #1: LOW LYING AREA BETWEEN TAXIWAYS WHISKEY AND BRAVO
8	GLYCOL TREATMENT PLANT
9	EXCELAIRE, LLC AND HAWTHORNE GLOBAL AVIATON SERVICES
10	SHELTAIR ISLIP, LLC
11	HERTZ/AVIS/BUDGET
12	WHITNEY HANGER
13	CENTRAL AVIATION AND MARINE CORPORATION (CAMCO)
14	COMPOSTING FACILITY
15	NEW YORK ARMY NATIONAL GUARD AVIATION UNIT

LEGEND:

- SITE BOUNDARY
- APPROXIMATE LIMITS OF AREA
- # ON SITE AREA ID
- EXISTING MONITORING WELLS
- PROPOSED VAP LOCATION
- GROUNDWATER FLOW DIRECTION (12/2022 SITE CHARACTERIZATION)
- 44 GROUNDWATER CONTOUR IN FEET (12/2022 SITE CHARACTERIZATION)

NOTES:

1. AERIAL PHOTOGRAPHY: 2019 MICROSOFT CORPORATION © 2019 DIGITAL GLOBE © CNES (2019) DISTRIBUTION AIRBUS DS BING.
2. PARCEL BOUNDARY DATA OBTAINED FROM SUFFOLK COUNTY, REAL PROPERTY TAX SERVICE AGENCY GEOGRAPHICAL INFORMATION SYSTEMS.

0 1000' 2000'

APPROXIMATE SCALE IN FEET

LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NEW YORK
REMEDIAL INVESTIGATION WORK PLAN

FACILITY WIDE SCOPE – GROUNDWATER

ARCADIS

FIGURE **11**

CITY:(Req'd) DIV/GROUP:(Req'd) DB:(Req'd) LD:(Opt) PIC:(Opt) PM:(Req'd) TM:(Opt) LYR:(Opt)ON=*;OFF=*REF*
C:\Users\mwasilewski\DCACCDocs\Arcadis ACC US\AUS-999999999-TOWN OF ISLIP_MACARTHUR AIRPORT_ISLIP_NY\Project Files\10_WIP\10T_ARC_ENV\2025\01-DWG\R1WP-F12 - FW_SCOPE - GW.dwg LAYOUT:12 SAVED: 5/14/2025 7:54 AM ACADVER: 24.2S (LMS TECH) PAGESETUP: ----
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Appendix A

List of Site Contacts

APPENDIX A – LIST OF SITE CONTACTS

Name	Phone/Email Address
Site Owner and Remedial Party: Town of Islip /Long Island MacArthur Airport, Robert A. Schneider, Commissioner of Aviation	631-467-3300 rschneider@islipny.gov
Qualified Environmental Professional: David Stern	516-369-7440 David.Stern@arcadis.com
Arcadis New York Professional Engineer: Christopher Engler, PE	315-409-6579 christopher.engler@arcadis.com
NYSDEC DER Project Manager: Jolene Lozewski	518-402-8805 Jolene.Lozewski@dec.ny.gov
NYSDEC DER Project Manager's Supervisor: Bob Corcoran	bob.corcoran@dec.ny.gov
NYSDEC Site Control: TBD	
NYSDOH Project Manager: Julia Kenney	julia.kenney@health.ny.gov
Remedial Party Attorney: Michael J. Cahill	631-588-8778 mjc@germanocahill.com
Remedial Party Attorney: Nick Rigano	631.756.5900 nrigano@riganollc.com
Remedial Party Attorney: William Garbarino	wgarbarino@islipny.gov

Appendix B

Field Sampling Plan (FSP)

Town of Islip

FIELD SAMPLING PLAN

Long Island MacArthur Airport

100 Arrival Ave, Ronkonkoma NY
NYSDEC Site No. 152251

May 19, 2026

FIELD SAMPLING PLAN

Long Island MacArthur Airport

100 Arrival Ave, Ronkonkoma NY

Site No. 152251

Nathan Nagle
Senior Scientist

Steven Feldman
Principal Scientist

David Stern
Project Manager

Prepared for:

Town of Islip

Long Island MacArthur Airport

100 Arrival Avenue

Ronkonkoma, NY 11779

Prepared by:

Arcadis of New York, Inc.

Two Huntington Quadrangle

Suite 1S10

Melville

New York 11747

Tel 631 249 7600

Fax 631 249 7610

Our Ref.:

30166065.00003

Date:

May 2026

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7 Waste Management and Disposal 7-12

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ATTACHMENTS

Attachment A-1 – Standard Operating Procedures

- TGI – PFAS Field Sampling Guide. September 2023.
- TGI – Soil Description. June 2024.
- TGI – Soil Drilling and Sample Collection. April 2023.
- TGI – Vertical Aquifer Profiling. June 2022.
- TGI – General Slug Testing. March 2024.
- TGI – Standard Groundwater Sampling for Monitoring Wells. April 2022.
- TGI – Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells. April 2023.
- TGI – PFAS-Specific Drilling and Monitoring Well Installation. August 2020.
- TGI – Monitoring Well Development. April 2023.
- TGI – Investigation-Derived Waste Handling and Storage. May 2020.

ACRONYMS AND ABBREVIATIONS

bls	below land surface
CAMP	Community Air Monitoring Plan
COC	Chain-of-Custody Form
DO	dissolved oxygen
DOT	Department of Transportation
FS	Feasibility Study
FSP	Field Sampling Plan
ft	feet
GIS	geographic information system
GPS	global positioning system
HASP	Health and Safety Plan
HDPE	high-density polyethylene
IDW	investigation-derived waste
ISP	Long Island MacArthur Airport
NYSDEC	New York State Department of Environmental Conservation
ORP	oxidation-reduction potential
PFAS	per-and polyfluoroalkyl substances
PFOS	perfluorooctanesulfonic acid
PID	photoionization detector
PPE	personal protective equipment
PVC	polyvinyl chloride
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RI	Remedial Investigation
RIWP	Remedial Investigation Work Plan
SCWA	Suffolk County Water Authority
SCR	Site Characterization Report
Site	Long Island MacArthur Airport
SOP	standard operating procedure

Town	Town of Islip
USEPA	U.S. Environmental Protection Agency
VAP	vertical aquifer profiling
VOC	volatile organic compound

1 PROJECT BACKGROUND

The Long Island MacArthur Airport (ISP or Site) is located at 100 Arrival Avenue in the hamlet of Ronkonkoma, Town of Islip (Town), New York. According to the Order on Consent (Index R1-20180228) (New York State Department of Environmental Conservation [NYSDEC] 2019) and the Suffolk County geographic information system (GIS) Viewer, the Site is identified as Parcel 500-106-1-6.9 and is owned by the Town. The Site is utilized as a municipal airport serving both commercial and general aviation flights and consists of a complex of buildings and structures positioned on approximately 1,141.8 acres. The Site is bounded to the north by the Long Island Railroad facility and Railroad Avenue; to the east by Smithtown Avenue with mixed commercial and industrial use; to the west by Lincoln Avenue with mixed residential and commercial use; and to the south by Veterans Memorial Highway along with mixed commercial and industrial use.

The Site was first developed in the late 1940's, prior to this date the Site was undeveloped grassland. Former Site operations are detailed in the Records Search Report (Arcadis 2019). The Site has been reclassified as a Class 2 site, indicating the presence of hazardous waste posing a significant threat to public health or environment.

Following finalization of the Site Characterization Report (SCR), the NYSDEC issued an Order on Consent (Index R1-20180228; the Order; NYSDEC 2024) to the Town. The Order was executed between the Town and the NYSDEC on February 15, 2024, with an effective date of February 25, 2024. The Order required that a Remedial Investigation (RI)/Feasibility Study (FS) be conducted for the Site. The Remedial Investigation Work Plan (RIWP) follows the findings of the SCR (Arcadis 2022).

2 INVESTIGATION ACTIVITIES

In general, sample collection efforts during investigation activities include:

- Collection of soil samples from soil borings
- Measurement of water levels in monitoring wells
- Collection of groundwater samples from vertical aquifer profiling (VAP) borings and permanent monitoring wells
- Collection of investigation-derived waste (IDW) liquid and solid media samples for waste characterization purposes

3 PRE-FIELD PREPARATION AND EQUIPMENT

The following sections describe the preparation that will be performed and equipment that is needed prior to mobilization to the field to conduct the activities specified in the Field Sampling Plan (FSP). The field project team (technicians, scientists, and engineers) will be responsible for obtaining, operating, and maintaining the required equipment, collecting the samples as specified herein, and for procuring and

maintaining sample containers pertinent to the collection of environmental samples. The following text describes these procedures in detail.

In general, the pre-cleaned environmental sample containers (bottles) will be provided by the analytical laboratory in accordance with procedures and requirements set forth in the Quality Assurance Project Plan (QAPP). The sample containers will be inventoried and inspected prior to work to verify that the required containers are present and in good condition. Specific sample container inspection procedures are as follows:

- Water and soil sample bottles will be inventoried and inspected to ensure that the required bottles are present, visually clean, unbroken, and have been properly preserved by the laboratory (see QAPP).

Field equipment will be inventoried and inspected by the field team members performing the work. The equipment listed below will be available and used during field activities:

- Personal protective equipment (PPE) and air monitoring equipment, as defined in the Health and Safety Plan (HASP) and Community Air Monitoring Plan (CAMP)
- Health and safety forms, as specified in the HASP
- Field daily logs or bound logbook or electronic recordkeeping, as specified in the QAPP
- Weighted tape measure, accurate to one hundredth of one foot
- Powdered Alconox
- Teflon-free scrub brushes
- Distilled/de-ionized water
- Per-and polyfluoroalkyl substances (PFAS)-free water provided by laboratory
- 4-millimeter-thick plastic sheeting

The following additional equipment/forms shall be used during collection of soil samples for lithologic characterization and/or laboratory analysis:

- Soil Sample/Core Logs, calibration logs, chains of custody (as specified in the QAPP), and Arcadis Soil Description Standard Operating Procedure (SOP) (**Attachment A-1**)
- Portable table for logging soil samples
- New, plastic 5-gallon buckets for sampling tool decontamination (decontamination procedures are provided in Section 6)
- Hand auger, stainless steel spoon, trowel and bowls for surface soil sample collection
- Plastic coolers for sample preservation, storage, and shipment
- Soil sample containers (dependent on analysis performed, refer to the QAPP)

The following additional equipment/forms shall be available and used during collection of groundwater samples from temporary and permanent monitoring wells:

- Water Sampling logs, calibration logs, chains of custody, as specified in the QAPP
- Sampling pumps, as specified by well type:
    - 2-inch diameter PFAS-free bladder pump, oil-free portable air compressor and controller (e.g., QED Well Wizard), regulator and pneumatic tubing
    - Submersible pump, portable generator and controller, regulator and tubing
    - 2-inch high-density polyethylene (HDPE) bailer

- Electronic PFAS-free water-level indicator, accurate to one hundredth of one foot
- Portable field instruments including pH, conductivity, temperature, turbidity, and the associated calibration standards
- Purge water flow-through cell
- Purge water containers (New York State Department of Transportation-compliant 55-gallon capacity close-top drums [water] or open-top drums [soil cuttings/solids])
- Plastic coolers for sample storage and shipment
- Disposable in-line field filters (0.45 micron) for filtering of samples for analysis of dissolved-phase inorganic parameters (e.g., metals)
- New 5/8-inch inner-diameter HDPE tubing
- New, disposable, 2-inch diameter HDPE bailers and cotton or nylon rope
- Water sample containers (to be provided by analytical laboratory and are dependent on analysis performed, refer to the QAPP)

The following equipment shall be used during the collection of stormwater grab samples:

- Water Sampling Logs, calibration logs, chains of custody, as specified in the QAPP
- Portable field parameter instruments, including pH, conductivity, temperature, turbidity, spectrophotometer, and the associated calibration standards
- Water sample containers (to be provided by analytical laboratory and are dependent on analysis performed, refer to the QAPP
- 6- to 12-foot-long swing sampler
- An unpreserved sample transfer container will be used at each sample location
- Plastic coolers for sample preservation, storage, and shipment

The following equipment shall be used during collection of soil and sediment samples:

- Stainless steel hand auger
- Lexan or equivalent tubing
- Differential global positioning system (GPS) equipment
- Stainless steel bowl and trowel
- Plastic coolers for sample preservation, storage, and shipment

4 SAMPLING ASSOCIATED WITH INVESTIGATION ACTIVITIES

The following sections describe methods for sampling associated with investigation activities. The QAPP provides additional details regarding field records and Quality Assurance/Quality Control (QA/QC) samples, frequency and protocols, sample labeling, and sample custody.

4.1 Sample Locations

The locations of soil and groundwater sample locations are shown on the figures in the RIWP.

Underground and aboveground utilities will be identified prior to any drilling or subsurface sampling. Utilities will be located by contacting responsible agencies by phone (New York's DigNet of New York City & Long Island) so that their underground utilities can be marked at the Site. Utilities will be located and identified following the procedures outlined in the Site Health and Safety Plan. Three lines of evidence will be used ensure safety of intrusive work. Other potential on site hazards such as traffic and overhead hazards will be identified during a site reconnaissance visit. The work will be planned, in conjunction with Site occupants, to minimize impacts and promote safe conditions for Site operation, on site workers and Site occupants.

4.2 Sampling Procedures for PFAS

PFAS samples, comprised of soil and groundwater samples, will be collected as specified in the RIWP. PFAS can be introduced into the sample by common PFAS-containing materials that may be present at the sampling site. Therefore, avoiding cross-contamination from PFAS containing materials during this investigation will be of utmost importance.

Prior to drilling and sampling activities, equipment that will be in contact with samples will be reviewed to our best knowledge and checked against the PFAS Sampling TGI (**Attachment A-1**) to avoid PFAS containing material, to the extent practical. This equipment includes, but is not limited to, drilling equipment (i.e. drill rods, macro-cores, core barrels, Push-Ahead™ tools and well material), sampling equipment (tubing, purge pump, O-rings etc), field supplies (i.e. PPE, trowels/spoons and plastic sheeting).

Sections 4.3 through 4.8 discuss boring and sampling general procedures for soil and groundwater. For PFAS sample collection, procedures outlined in the **Attachment A-1** PFAS Sampling TGI will be followed.

4.3 Soil Borings

This section describes methods for drilling soil borings and collecting soil samples. Surface soil samples will be collected using a hand auger and subsurface soil samples drilled using direct-push or sonic drilling methods, depending on field conditions and work plan objectives.

When using direct-push methods, the Geoprobe® Dual Tube Sampling System will be used. The Geoprobe® Dual Tube Sampling will be used. The Geoprobe® Dual Tube Sampling System employs an outer casing and inner rod string assembly. Soil sampling will be conducted at the boring locations by advancing 3.25-inch outside diameter probe rods (outer casing) and a 5-foot-long sample liner. Dual tube sampling uses two sets of probe rods to collect continuous soil cores. One set of rods is driven into the ground as an outer casing. These rods receive the driving force from the hammer and provide a sealed borehole from which soil samples may be collected. The second, smaller set of rods (inner rod string) are placed inside the outer casing. The inner rod string holds the sample liner in place as the outer casing is driven over the sampling interval. The inner rod string is then retracted to retrieve the filled sample liner.

When using sonic drilling techniques, soil sampling will be conducted at the boring locations by driving a 5-foot long 4-inch diameter core barrel using vibration, rotation, and a downward force. Once the core barrel has been advanced, a 6-inch diameter secondary or "override" casing will be advanced down to the

same depth as the inner core barrel. The override casing keeps the borehole from collapsing while the inner core barrel is removed. Once the core barrel is removed, the soil core will be extruded from the core barrel.

Soil samples will be visually characterized for color, texture, and moisture content as described in the Arcadis Soil Description SOP (**Attachment A-1**). Soil samples will be collected following the procedures described in the Arcadis Soil Drilling and Sample Collection SOP (**Attachment A-1**) and Section 5 of this FSP. Soil samples will be analyzed for the compounds/analytes specified in the work plan.

Drilling and sampling of soil borings will include the following activities:

1. Determine location of the soil boring and avoid aboveground and underground utilities per the HASP.
2. Location will be recorded using hand-held GPS unit.
3. Drill rig will be mobilized to the proposed location.
4. Proposed location will be cleared of utilities to a depth of approximately 5 feet (ft) below land surface (bls) by hand digging or by air knife.
5. Drilling will commence and continuous soil cores will be collected.
6. Field geologist (in coordination with the driller) will monitor the formation drilled through and examine collected soil cores and cuttings for visual indication of impacts (photoionization detector [PID] readings above background, staining, or odor).
7. Soil samples will be collected for the specified analyses following procedures below:
    - Immediately after opening the sampler, obtain an organic gas measurement via PID, observe the geology of the soil cores and record observation on Soil Sample/Core Log;
    - Collect sample for volatile organic compound (VOC) analysis first.
    - Once the VOC container has been filled, the remaining soil should be thoroughly mixed in a decontaminated stainless-steel bowl. Once mixed, the soil should be placed in the remaining sample containers.
    - Label and return the sample container to the cooler.
    - If reusable, decontaminate the sampling equipment.
8. Boreholes will be backfilled per NYSDEC DER-10 Technical Guidance for Site Investigation and Remediation.

4.4 Vertical Aquifer Profiling

This section describes the methods to collect groundwater samples via VAP borings. VAP locations will be drilled using either sonic or rotary drilling methods. Groundwater samples will be collected at 20-foot sample intervals to an estimated total depth between 150 and 300 ft bls depending on location at the Site.

Sonic or rotary drilling methodologies will be utilized for VAP boring advancement and grab sampling will be completed using top-down, dual-tube (or equivalent) methods in accordance with the TGI for VAP for

PFAS Analysis (**Attachment A-1**) to reduce potential for cross-contamination between sampling intervals. The rig system will use a 4-inch core barrel with 6-inch outer casing to seal off the borehole.

Once the target interval is reached, a packer isolation groundwater profiler will be utilized for VAP groundwater sampling. The procedure will begin by driving the casing over the soil core, retrieving the soil core barrel, and then lowering a stainless screen and packer assembly to the base of the casing. The outer casing will then be extracted to expose the screen to the formation, and the packer will be inflated within the casing above the screen to isolate the screened interval from any water that might be above the packer in the sonic casing. A groundwater sample will then be collected from the discrete screened interval.

Soil cores collected during drilling will be logged for lithological descriptions at each location by the field geologist. A PID will be used to screen the breathing zone during boring activities. Observations and measurements will be recorded on field forms and in a non-treated field logbook. Photographs of the boring cores will also be taken, if permitted. At a minimum, depth interval, recovery thickness, PID concentrations, moisture, relative density, color, and texture will be recorded. Refer to the TGI for Soil Description and TGI for Soil Drilling and Sample Collection (**Attachment A-1**) for additional details.

Groundwater from VAP samples will be analyzed for PFAS using ASTM D8421 to provide rapid data and inform decisions in the field regarding the locations and construction of new monitoring wells. Groundwater from monitoring well samples will be analyzed for PFAS by United States Environmental Protection Agency (USEPA) Method 1633 to verify that NYSDEC data quality objectives have been met and to obtain data for risk assessment purposes. Groundwater samples will be collected in accordance with the procedures presented in Worksheet #14 and further detailed in the TGI for PFAS-Specific Drilling and Monitoring Well Installation (**Attachment A-1**).

4.5 Monitoring Well Drilling, Installation and Development

This section describes the methods to drill, install, and develop monitoring wells. The monitoring wells will be drilled using direct-push or sonic drilling methods depending on target depth. After completion of drilling and well installation, monitoring wells will be developed to establish hydraulic connection between the well and the formation. Monitoring wells will be installed using the procedures described in the Arcadis Monitoring Well Installation SOP (**Attachment A-1**).

4.5.1 Monitoring Well Drilling

Drilling and geological logging methods for monitoring well installation are as follows:

- Boreholes will be drilled using direct-push or sonic methods to depths as discussed in the work plans
- Continuous soil cores will be retrieved for field screening. Designated field geologist will log borehole geology and monitoring well specifications in the field book and/or field forms
- Soil cuttings will be placed in open-top drums supplied by the drilling subcontractor and containerized at the end of each workday

4.5.2 Monitoring Well Specifications

Monitoring wells will be installed according to the following specifications:

- 4-inch diameter threaded, flush-joint Schedule 40 polyvinyl chloride (PVC) casing and 10-foot long, 20 slot (0.020-inch) stainless steel screens will be installed.
- A 4-inch diameter, Schedule 40 PVC sump, minimum 1 foot in length, may be attached to the bottom of the screen for sediment accumulation and periodic removal as needed.
- The annular space around the screens will be backfilled with Morrie #1 (or equivalent) sand pack to a height of 1 to 2 ft above the top of the screen.
- A 2-foot-thick bentonite seal (pellets or slurry depending on depth of seal) will be placed above the sand pack. The bentonite seal will be allowed to hydrate before placing grout above the seal.
- The remainder of the annular space will be filled with a cement/bentonite grout to approximately 2 ft bls. The grout will be placed from the bottom up using a tremie pipe. The grout will consist of a cement mixture of one 94-pound bag of Portland cement, approximately 5 pounds of granular bentonite, and approximately 7 gallons of water. The grout will be allowed to set for a minimum of 48 hours before wells are developed if the grout is placed below the water table.
- Monitoring wells will be completed with a 10-inch diameter water-tight flush-mount or stick-up protective casing. A 2-foot by 2-foot cement pad will be installed around the flush-mount protective casing. The well casing will extend to within approximately 2 to 4 inches below land surface and will include an expandable, locking cap.
- The north side of the top of the PVC well casing will be marked (i.e. measuring point) and the elevation of the measuring point will be surveyed to the nearest 0.01 ft.

4.5.3 Monitoring Well Development

A minimum of 48 hours after installation, or the day after installation if the grout is located above the water table, monitoring wells will be developed by surging and pumping using the procedures outlined in the Arcadis Monitoring Well Development SOP (**Attachment A-1**). Development water will be containerized in drums to be provided by the drilling subcontractor. The wells will be developed until the turbidity is reduced to 50 nephelometric turbidity units or less or until the pH and specific conductivity measurements have stabilized. Following development, monitoring wells will be allowed to equilibrate for a minimum of two weeks before groundwater sampling is conducted. Monitoring well development will be overseen by a qualified person and the duration, method of development, and approximate volume of water removed will be recorded in a well development log and well construction log.

4.6 Hydraulic Measurements in Monitoring Wells

Hydraulic (i.e., water-level) measurements will be collected using the procedures described below:

1. For each monitoring well location, total well depth will be measured to ensure that the well screen is open to the formation. Then water-level measurements will be collected by measuring the depth to groundwater from the surveyed measuring point.

2. The water-level measurements will be made to the nearest one-hundredth of one foot with a decontaminated electronic water-level indicator.

3. The electronic water-level indicator or interface meter will be decontaminated between well locations using the methods described in Section 5 of this FSP.

4. Water-level and fluid-level measurements and other pertinent information (e.g., well designation) will be recorded as outlined in the QAPP.

Measurements will be made in as short a timeframe as practical to minimize temporal fluctuations in hydraulic conditions. A round of water-level measurements will be performed in conjunction with each groundwater sampling event, as discussed in the work plan.

4.7 Monitoring Well Groundwater Sample Collection

This section describes the methods to collect groundwater samples from monitoring wells. Groundwater investigation objectives will be described in the work plan. Groundwater samples will be analyzed for the analytes specified in the work plan and the QAPP. Additional details on performing groundwater sample collection are described below. Monitoring wells will be sampled at least one week after development.

General pre-sampling activities that will be performed during monitoring well groundwater sample collection include accessing the well, preparing the well for purging and sampling, and collecting initial measurements. To access the well, the protective casing will be unlocked, and surficial soil will be cleaned from around the wellhead if necessary. Plastic sheeting will be placed around the well and secured at the corners. The depth to water in the well will be measured to the nearest one-hundredth of one foot with an electronic water-level indicator or interface meter and the total depth of the well will be measured.

The monitoring wells will be purged and sampled using low-flow groundwater sampling procedures described in the ARCADIS Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells SOP (**Attachment A-1**). Monitoring wells will be purged and sampled using a decontaminated, Teflon-free bladder pump following USEPA low-flow (minimal drawdown) groundwater sampling procedures (EPA 1996) as follows:

- HDPE tubing will be connected to the pump. The pump and tubing will be gradually lowered to place the pump intake within the approximate center of the well screen zone or the midpoint of the saturated portion of the screen if the well bridges the water table.

- Groundwater will then be extracted from each well using low-flow techniques and will be directed into a flow-through cell. The flow-through cell will contain the dissolved oxygen (DO), oxidation-reduction potential (ORP), pH, conductivity, and temperature probes. Turbidity will be measured external to the flow-through cell. The wells will be purged at rates that do not exceed 500 milliliter per minute. Ideally, the pump rate should cause little water-level drawdown in the well (less than 0.3 ft and the water level should stabilize). The water level should be monitored every five minutes (or as appropriate) during pumping. Care should be taken not to cause the pump suction to be broken or entrainment of air in the sample. Record pumping rate adjustments and depths to

water. Pumping rates should, if needed, be reduced to the minimum capabilities of the pump to avoid pumping the well dry and/or to ensure stabilization of indicator parameters. If the recharge rate of the well is very low, purging should be interrupted so as not to cause the drawdown within the well to advance below the pump. However, a steady flow rate should be maintained to the extent practicable. Sampling should commence as soon as the volume in the well has recovered sufficiently to permit sample collection.

- Field parameters (DO, ORP, pH, conductivity, temperature, and turbidity) will be measured initially and at approximately five-minute intervals during purging and will be recorded on Low-Flow Groundwater Sampling Logs. Field parameters will be monitored with calibrated meters that will be calibrated daily according to the manufacturer's instructions. Completion of purging, and therefore, the actual volume of water purged from each well, will be based on the stabilization protocols described in the low-flow method.

- Following stabilization of field parameters as specified by the USEPA low-flow method, the groundwater samples will be collected directly from the pump discharge.

- Once sampling is complete, the pump will be gradually removed from the well and dedicated sampling equipment (e.g., tubing) will be disconnected from the pump and secured inside the well casing. The well will be closed and locked and the pump will be decontaminated.

4.8 Investigation-Derived Waste Sampling

IDW will be managed in accordance with the USEPA Interim Guidance on the Destruction and Disposal of PFAS and Materials Containing PFAS (USEPA 2024). IDW generated from investigation activities (i.e., soil borings/monitoring well installations, sampling of soil/groundwater and decontamination of associated equipment, or well abandonment) will be managed at a central staging location adjacent to Area 08: Glycol Treatment Plant consistent with the SC field program and then disposed in accordance with state guidelines and the TGI for Investigation-Derived Waste Handling and Storage (**Attachment A-1**). IDW will be separated by PPE and spent disposable sampling materials, aqueous waste, and solid waste. Aqueous and solids will be sampled as composites for characterization according to the disposal facility requirements before disposal at an off-site permitted facility. Arcadis will coordinate with ISP and the disposal contractor to arrange for signatures of waste manifests and for compliance with facility generator requirements. The final IDW disposal documentation will be included in the RI report.

5 SAMPLE COLLECTION, LABELING, HANDLING, AND ANALYSIS

This section describes sample collection, labeling, handling, and analysis.

5.1 Soil Samples

Soil sampling activities to be conducted includes the collection of soil samples from soil borings. Soil samples will be collected from the appropriate two-foot interval using a decontaminated stainless-steel spoon or trowel. The VOC sample will be immediately transferred directly into the laboratory-supplied sample bottles. A sufficient amount of the remaining soil will be homogenized by mixing the sample in a decontaminated stainless-steel bowl with a decontaminated stainless-steel spoon or trowel. Laboratory-supplied sample containers for other analytes (e.g., semi-volatile organic compounds, pesticides, polychlorinated biphenyls, metals) will then be filled. Sample bottle caps will be secured snugly, but not over-tightened.

Soil cores will be screened for organic vapors using the jar headspace method and a PID. In addition, a geologist will be onsite during the drilling operations to describe each sample in accordance with Arcadis Soil Description SOP (**Attachment A-1)**, and will include:

- Depth
- Sample recovery
- Soil type and sorting
- Color (natural color, discoloring due to impacts should be described as staining)
- Moisture content (dry, moist, wet)
- Texture
- Grain size and shape
- Relative density
- Consistency (degree of plasticity for fine-grained soils; viscosity for non-aqueous phase liquids such as taffy-like, etc.)
- Representativeness
- Miscellaneous observation (obvious odors – e.g., faint odor, moderate odor, strong odor, etc.)

Samples (including QA/QC samples specified in the QAPP) will be properly labeled and identified, and the Sample/Core Log and the Chain-of-Custody Form will be completed. The QAPP provides additional details regarding Field Records and QA/QC samples, frequency and protocols, and sample custody. Sample containers will be checked for proper identification/labeling and compared to the Chain-of-Custody Form (COC) for accuracy prior to packaging the sample for shipment. The COC will be placed in a sealed plastic bag and taped to the underside of the cooler lid. The sample containers will be wrapped with a cushioning material to preclude sample container breakage during shipment and placed in a cooler. Bagged ice will be placed in the cooler to keep the soil samples at 6 degrees Celsius until arrival at the laboratory. The cooler will be sealed with packaging tape and custody seals will be placed in such a manner that any opening of the cooler prior to arrival at the laboratory can be visually detected.

Samples will be delivered by overnight carrier or laboratory courier to the analytical laboratory following sample custody requirements specified in the QAPP. The laboratory will be prepared to receive the samples and perform preliminary extractions or analyses within the analytical method recommended holding times.

5.2 Groundwater Samples

Groundwater samples will be collected directly into the laboratory-supplied sample bottles. The flow of water from the sampling equipment will be adjusted to ensure slow, laminar flow so that no entrained air bubbles are present in VOC samples. Special care will be taken in filling and capping volatile organic analysis (VOA) vials so that headspace/air bubbles are not present in the groundwater samples. In addition, overflowing bottles will be avoided to prevent the loss of floating substances or preservatives that may have already been added to the bottle. All sample bottle caps will be secured snugly, but not over-tightened.

The groundwater samples will be described for any visual observations of impacts, odors, or coating of sampling equipment. This information will be recorded on the Water Sampling Log or Low-Flow Groundwater Sampling Log.

Samples (including QA/QC samples specified in the QAPP) will be properly labeled and identified, and the Low-Flow Groundwater Sampling Log and Chain-of-Custody Form will be completed. The QAPP provides additional details regarding Field Records and QA/QC samples, frequency and protocols, sample labeling, and sample custody. Sample containers will be checked for proper identification/labeling and compared to the COC for accuracy prior to packaging any sample for shipment. The COC will be placed in a sealed plastic bag and taped to the underside of the cooler lid. The samples will then be wrapped with a cushioning material to preclude sample container breakage during shipment and placed in a cooler. Bagged ice will be placed in the cooler to keep the groundwater samples at 6 degrees Celsius until arrival at the laboratory. The cooler will be sealed with packaging tape and custody seals will be placed in such a manner that any opening of the cooler prior to arrival at the laboratory can be visually detected.

Samples will be delivered by overnight carrier or laboratory courier to the analytical laboratory following sample custody requirements specified in the QAPP. The laboratory will be prepared to receive the samples and perform preliminary extractions or analyses within the analytical method recommended holding times.

6 FIELD DECONTAMINATION PROCEDURES

Decontamination procedures for non-dedicated field equipment are presented in detail in this section and include decontamination procedures associated with non-dedicated sampling equipment and downhole drilling tools and equipment. In general, after decontamination is completed, items will be stored in a manner to preserve their decontaminated condition prior to use.

6.1 Drilling Equipment Decontamination

A decontamination pad will be constructed and lined with plastic sheeting. Drilling equipment including the rear-end of the drilling rig (if necessary), sampling tools, bits, drill rods, and tremie pipe will be cleaned on the decontamination pad with a high-pressure hot water "steam cleaner" unit and scrubbed with a wire brush, as needed, to remove foreign material (e.g., soil, oil etc.). Equipment will be decontaminated prior

to the start of drilling, between each borehole, and prior to leaving the Site. Tools, drill rods, and other downhole equipment will be placed on sawhorses, decontaminated pallets, or polyethylene plastic sheets following steam cleaning. Direct contact with the ground will be avoided. Decontamination water will be pumped into and containerized in DOT-approved 55-gallon open-top steel drums. Open-top drums will remain closed when not in use.

Following decontamination of Site equipment, the decontamination pad will be decommissioned. The decommissioning will be completed by:

- Transferring the bulk of the remaining liquids and solids into the drums or tanks to be provided by the drilling subcontractor.
- Rolling the sheeting used in the decontamination pad onto itself to prevent discharge of the remaining materials to the ground surface. Once rolled up, the polyethylene sheeting will be placed in the drums used for disposal of PPE and disposable equipment.

6.2 Sampling Equipment Decontamination

Soil sampling, groundwater sampling, and sediment sampling equipment requiring decontamination includes, but is not limited to, Push-Ahead™ samplers, stainless steel spoons/trowels/bowls, interface meters, non-dedicated pumps/appurtenances, macro cores, core barrels and drill rods. The sampling equipment will be washed with potable water and a detergent (powdered Alconox) to remove foreign material. Decontamination may be conducted at the sampling location if liquids are contained in pails or buckets. The sampling equipment will then be rinsed with potable water and a final distilled water rinse. The equipment will be decontaminated before and between each use and prior to demobilization. Washed equipment will not be placed directly on the ground. Decontaminated equipment will either be used immediately or wrapped in plastic for storage or transportation from the designated decontamination area to the sampling location.

Water quality probes and water-level indicators will be decontaminated by rinsing with distilled water. Decontamination fluids will be containerized prior to off-site transportation and disposal as described in Section 7 of this FSP.

Disposable tubing from groundwater sampling will be discarded as general trash after each use.

7 WASTE MANAGEMENT AND DISPOSAL

Solid and liquid IDW generated during field activities including, but not limited to, drill cuttings, monitoring well purge water, and decontamination water will be managed and disposed as outlined in this section. IDW will be containerized in appropriate waste containers and staged on site at a central location prior to off-site disposal, following the procedures described in the ARCADIS Investigation-Derived Waste Handling and Storage SOP (**Attachment A-1**). Soil cuttings, PPE, and spent disposable sampling materials will be segregated by waste type and placed in DOT-approved 55-gallon steel drums. Decontamination water and development water will be stored in DOT-approved 55-gallon steel drums or a polyethylene storage tank. Storage vessels will be appropriately labeled with the contents, generator, location, and date for later off-site transportation and disposal by the Site.

8 REFERENCES

Arcadis. 2019. Records Search Report. Long Island MacArthur Airport. 100 Arrival Ave, Ronkonkoma NY. NYSDEC Site No. 152251.

Arcadis. 2022. Site Characterization Report. Long Island MacArthur Airport. 100 Arrival Ave, Ronkonkoma NY. NYSDEC Site No. 152251.

NYSDEC. 2019. Order on Consent and Administrative Settlement, site # 152251.

NYSDEC. 2024. Order on Consent and Administrative Settlement, site # 152251.

ATTACHMENT A-1

Standard Operation Procedures

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Appendix C

Quality Assurance Project Plan (QAPP)

Appendix C

Quality Assurance Project Plan (QAPP)

Town of Islip

Quality Assurance Project Plan

Long Island MacArthur Airport

100 Arrival Ave., Ronkonkoma, New York

Site No. 152251

February 2024

Quality Assurance Project Plan

Town of Islip
Long Island MacArthur Airport
100 Arrival Ave., Ronkonkoma, New York
Site No. 152251

February 2024

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Approval Page

This Quality Assurance Project Plan (QAPP) has been prepared by Arcadis of New York, Inc. (Arcadis) for the Town of Islip, Long Island MacArthur Airport sampling program. This plan has been reviewed and approved by the project management team and shall be followed by all personnel working on the environmental sampling activities.

Approvals are indicated as follows:

David Stern **Project Manager** **Arcadis of New York, Inc.**	 8/22/24
	Signature, Date
Jennifer Singer **Quality Assurance Manager** **Arcadis of New York, Inc.**	 8/22/2024
	Signature, Date

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Acronyms and Abbreviations

AOPC	area of potential concern
Arcadis	Arcadis of New York, Inc.
ASP	Analytical Services Protocol
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
DQI	data quality indicator
DQO	data quality objective
DUSR	Data Usability Summary Report
EDD	electronic data deliverable
EIS	extracted internal standard
FSP	Field Sampling Plan
HASP	Health and Safety Plan
IRM	Interim Remedial Measure
LCS	laboratory control sample
LLLCS	low level laboratory control sample
MDL	method detection limit
MS/MSD	matrix spike/matrix spike duplicate
NIS	non-extracted internal standard
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
OSHA	Occupational Safety and Health Administration
PCB	polychlorinated biphenyl
PFAS	per- and polyfluoroalkyl substances
QA	quality assurance
QAM	quality assurance manager
QAPP	Quality Assurance Project Plan
QC	quality control
QP	Quality Procedure
RI/FS	Remedial Investigation/Feasibility Study
RL	reporting limit
RPD	relative percent difference

RSR	Records Search Report
SC	Site Characterization
SCWA	Suffolk County Water Authority
SDG	sample delivery group
SOP	Standard Operating Procedure
SVOC	semivolatile organic compound
TAL	target analyte list
TCL	target compound list
TGI	Technical Guidance Instruction
TOC	total organic carbon
Town	Town of Islip
USDOT	United States Department of Transportation
USEPA	United States Environmental Protection Agency
VAP	vertical aquifer profiling
VOC	volatile organic compound

A. Group A: Project Management and Information/ Data Quality Objectives

A.4. Project Purpose, Problem Definition, and Background

A.4.1 Project Purpose

This QAPP is prepared as part of the On-Site Remedial Investigation/Feasibility Study Work Plan (RI/FS Work Plan) for the Long Island MacArthur Airport located at 100 Arrival Ave in Ronkonkoma, New York (the Site). This QAPP presents the sampling and analytical methods and procedures that will be used during the RI/FS at the site. This QAPP is prepared on behalf of Town of Islip (the Town).

This QAPP satisfies United States Environmental Protection Agency (USEPA) requirements for a QAPP, in accordance with the following guidelines:

- USEPA, "Quality Assurance Project Plan Standard," CIO 2105-S-02.0, July 2023 (USEPA 2023b).
- USEPA, "Guidance on Systematic Planning Using the Data Quality Objectives Process", EPA-QA/G-4, February 2006 (USEPA 2006).

A.4.2 Problem Definition

The Site is presently classified as Class "2" in the New York State Superfund Registry of Inactive Hazardous Waste Disposal Sites. The Class "2" listing followed the submission of the Records Search Report and Site Characterization Report to the New York State Department of Environmental Conservation (NYSDEC). Following finalization of these documents, the NYSDEC issued Order on Consent (Index R1-20180228) to the Town. The Order required that an RI/FS be conducted for the Site.

The purpose of the field program that is the subject of this QAPP is to collect soil samples from soil borings and groundwater samples from vertical aquifer profile (VAP) borings and groundwater monitoring wells to support the first phase of the remedial investigation. Specifically, the investigation focuses on the investigation of on-site areas of potential concern (AOPCs) retained from the Site Characterization and other areas requested for additional investigation by NYSDEC (herein referred to as "Areas"). In total, nine Areas will be investigated.

A.4.3 Background

A.4.3.1 General Site Description

The Site is located at 100 Arrival Avenue in the hamlet of Ronkonkoma, Town of Islip, New York. According to the Order on Consent and the Suffolk County GIS Viewer, the Site is identified under parcel number 500-106-1-6.9 and is owned by the Town of Islip. The Site is utilized as a local airport serving both commercial and general aviation flights per the Records Search Report (Arcadis, 2019). The Site consists of a complex of buildings and structures positioned on approximately 1,208 acres. The Site is bounded to the north by the Long Island Railroad facility and Railroad Avenue; to the west by Smithtown Avenue with mixed commercial and industrial use; to the east by Lincoln Avenue with mixed residential and commercial use; and to the south by Veterans Memorial Highway with mixed commercial and industrial use (Arcadis, 2019).

A.5. Project Task Description

Sampling protocols to be followed during investigation activities are detailed in the Field Sampling Plan (FSP) (Appendix B of the RI/FS Work Plan). The investigation activities will include soil borings; soil sampling and groundwater sampling; sediment sampling; storm water sampling; performance of hydraulic monitoring and a comprehensive groundwater sampling round including existing monitoring wells and new monitoring wells installed during the investigation activities. The objective of the sample collection is to develop an approach and methodology to gather information necessary to characterize whether releases have occurred at the Site, and to determine whether or not those releases pose a potential threat to public health and the environment.

Soil samples will be collected at each Area to characterize and delineate per- and polyfluoroalkyl substances (PFAS) impacts, support fate and transport evaluation, and determine potential risk associated with surficial soils. Soil samples will be analyzed for PFAS (ASTM D8421 and USEPA 1633). Method ASTM D8421 will provide rapid data and inform potential step-out boring locations. These results will be supplemented by USEPA 1633 analysis to provide data appropriate for risk assessment purposed. A limited number of samples will also be analyzed for target compound list (TCL) volatile organic compounds (VOCs; USEPA method 8260C), semi-volatile organic compounds including 1,4-dioxane (SVOCs; USEPA method 8270D), polychlorinated biphenyls (PCBs; USEPA method 8082B), pesticides (USEPA method 8081B), and herbicides (USEPA method 8151A); target analyte list (TAL) metals (USEPA method 6010C) and mercury (USEPA method 7471B); and hexavalent chromium (USEPA method 7196A). Additionally, total organic carbon (TOC), pH, and grain size/clay content will also be analyzed in one soil sample per Area.

Groundwater samples will be collected to assess the nature, extent, and transport of PFAS in groundwater and inform additional phase(s) of remedial investigation work not discussed in the RIWP that may include off-site locations. Groundwater samples will be analyzed for PFAS (ASTM D8421 and USEPA 1633). Groundwater from VAP samples will be analyzed for PFAS using ASTM D8421 to provide rapid data and inform decisions in the field regarding the locations and construction of new monitoring wells. These results will be supplemented by USEPA 1633 analysis to provide data appropriate for risk assessment purposed. A limited mumber of groundwater samoples which is equivalent to 10% of the groundwater samples and representing all AOPCs and site-wide areas, will undergo full suite analysis.

Tables 1a and 1b presents constituents of each analytical method that will be analyzed for in soil and groundwater samples, along with screening levels and laboratory reporting limits.

Health and safety protocols to be followed by field personnel during completion of the site characterization investigation activities are discussed in the Health and Safety Plan (HASP) (Appendix E of the RIWP).

The schedule for the activities discussed in this QAPP is included in Table 4 of the RI/FS Work Plan.

A.6. Information/Data Quality Objectives and Performance/ Acceptance Criteria

A.6.1 Data Quality Objectives

The data quality objective (DQO) process outlined below establishes performance or acceptance criteria, which serve as the basis for designing the plan for collecting data of sufficient quality and quantity to support the goals

of this investigation. The DQO process, consisting of seven steps, was used to establish the final data collection design.

A.6.1.1. Step 1: Problem Statement

Following finalization of the Site Characterization Report, the NYSDEC issued an Order on Consent to the Town, requiring that an RI/FS be conducted for the Site.

A.6.1.2. Step 2: Identify the Goals of the Investigation

The general objectives of the NYSDEC RI/FS process are as follows:

- Determine the nature and extent of PFAS and assess potential impacts to receptors, including public health, welfare, and the environmental from the release or potential release of PFAS at or from the Site.
- Determine the nature and extent of TCL and TAL parameters in soil and groundwater as requested by NYSDEC even though they are not considered constituents of concern at the Site.
- Develop and evaluate alternatives for remedial action (if needed) to prevent, mitigate, and otherwise respond to or remedy a release or potential release of PFAS at or from the Site be conducting an FS.
- Compile all related (i.e., current and previous) data from NYSDEC, Suffolk County Water Authority (SCWA), and the Town of Islip into one comprehensive RI report.
- Evaluate the need for an Interim Remedial Measure (IRM) and, if needed, evaluate potential technologies for and propose an IRM.
- Incorporate all elements of an RI/FS as set forth in the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, the USEPA Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA, and appropriate USEPA and NYSDEC technical and administrative documents.

A.6.1.3. Step 3: Identify Information Inputs

The activities of the RI include the following:

- Drilling and sampling of soil from soil borings.
- Drilling and sampling of soil and groundwater from VAPs and documenting borehole geophysics.
- Drilling, installing, and developing the new permanent monitoring wells.
- Performing at least one round of water level measurements from new and existing monitoring wells at the Site.
- Collecting at least one round of groundwater samples from new and existing monitoring wells at the Site.

A.6.1.4. Step 4: Define the Boundaries of the Investigation

The Areas in which soil and groundwater samples are proposed include the following:

- Area 1: Recharge Basin #1
- Area 2: Recharge Basin #2
- Area 3: Fire Training Area #1, Northeast Wooded Area
- Area 4: Fire Training Area #2, Northwest Behind Compost Facility

- Area 5: Equipment Cleanout Area by SCWA Tower
- Area 6: Old Fire House and New Firehouse (one location)Area 8: Glycol Treatment Plant
- Area 11: Hertz/Avis/Budget
- Area 16: Helicopter Crash Site
- Facility Wide: locations not directly associated with an Area, generally located downgradient of Areas or on the site boundary

The locations of the proposed soil and groundwater samples are included in the RI/FS Workplan.

A.6.1.5. Step 5: Develop the Analytic Approach

Soil and groundwater samples will be collected and analyzed to generate the necessary data for this investigation. Briefly, samples will be analyzed for the following parameters:

- PFAS by ASTM D8421 and USEPA 1633

Additionally, a limited number of soil samples will be analyzed for the following parameters:

- TCL VOCs by USEPA method 8260C
- TCL SVOCs including 1,4-dioxane by USEPA method 8270D
- TCL PCBs by USEPA method 8082B
- TCL pesticides by USEPA method 8081B
- TCL herbicides by USEPA method 8151A
- TAL metals by USEPA method 6010C, 7470A, and 7471B
- Hexavalent chromium by USEPA method 7196A

A.6.1.6. Step 6: Develop Performance or Acceptance Criteria

Two sources of decision errors have been identified: sampling design errors and measurement errors. Sampling design errors are a function of the selection of sample locations or analytical methods used to characterize the site to be studied. Measurement errors are a function of the procedures used to collect and analyze the samples. Decision errors will be limited by careful evaluation of data and adherence to established data collection procedures.

The sampling design for the investigation is judgment-based rather than probability-based. Consequently, statistical inference techniques to precisely quantify uncertainty are not applicable, and no statistical limits on uncertainty were specified. A judgmental sampling design is appropriate for investigations in which reliable historical and physical knowledge of the feature or condition already exists. The tolerable limits on decision errors associated with statistical sampling designs do not apply directly to judgmentally located samples. Sampling design error was controlled by careful planning consultation with stakeholders and using the professional judgment of project personnel to achieve the study goals.

Measurement errors that arise during the multiple steps of the sample measurement process (e.g., sample collection, sample handling, sample preservation, sample analysis, data reduction, and data handling) are possible regardless of the sample design. Neither measurement errors nor variability can be eliminated, but they can be measured and evaluated with appropriate procedures. Measurement error is further managed by using standard procedures, reviewing data records, and managing data quality.

Performance criteria specific to this QAPP are further described in **Section A.6.2**.

A.6.1.7. Step 7: Develop the Plan for Obtaining Data

The plan for obtaining the data is included in the RI/FS Work Plan.

A.6.2 Data Quality/Performance Indicators

The primary quality assurance objective is to ensure that the data are of known and acceptable quality, and that the analysis is performed with sufficient sensitivity to confidently detect target parameters. To support the overall project DQOs and to gauge measurement performance, six data quality indicators (DQI) have been defined: precision, accuracy, representativeness, comparability, completeness, and sensitivity. Each DQI is defined below.

Precision. Precision is a measure of agreement among repeated measurements. The goal is to maintain a level of analytical precision consistent with the project objectives. To maximize precision, sampling and analytical procedures will be followed and work will adhere to established protocols presented in this QAPP. Analytical precision will be determined through the use of the same analytical methods to perform repeated analyses on the same sample, including the analysis of matrix spike duplicate samples laboratory duplicates, and field duplicates. Collection of field duplicate samples allows for evaluating the precision of sample collection, sample handling, preservation, and storage.

Precision for laboratory and field measurements will be expressed as the relative percent difference (RPD) between two duplicate determinations. Acceptance criteria for laboratory precision are presented on **Table 2**. Calculation methods for determining precision are provided in **Section B.4.2**.

Accuracy/Bias. Accuracy is the degree of agreement of a measured value with its true value. Accuracy can be expressed as a percent recovery or percent deviation of the measurement with respect to its known or true value. Accuracy will be determined through evaluation of spike recoveries (e.g., surrogate compound recoveries, laboratory control sample (LCS) recoveries, and matrix spike and matrix spike duplicate (MS/MSD) recoveries) and instrument calibration. Acceptance for spike recoveries is presented in **Table 2**. Calculation methods for determining accuracy are provided in **Section B.4.3**.

Bias is defined as the constant or systematic distortion of a measurement process as a persistent positive or negative deviation from the known or true value. This may be due to improper sample collection, sample matrix, poorly calibrated analytical or sampling equipment, or limitations or errors in analytical methods and techniques. Blank samples (laboratory or field) are also used to assess contamination of samples that may bias results high.

Representativeness. Representativeness is the extent to which measurements represent the site conditions. It is dependent on sampling and analytical variability, and the variability (or homogeneity) of the site itself. The sampling activities have been designed to assess the presence of the constituents at the time of sampling. This QAPP presents field sampling methodologies and laboratory analytical methodologies. The use of the prescribed field and laboratory analytical methods with associated holding times and preservation requirements are intended to provide representative data.

Comparability. Comparability is defined as the extent to which data from one data set can be compared directly to similar or related data sets and/or decision-making standards. Comparability between this investigation and to the extent possible, with existing data will be maintained through consistent use of the sampling and analytical methodologies set forth in this QAPP, the analytical methods, stringent applicable of established quality assurance (QA)/quality control (QC) procedures, and utilization of appropriately trained personnel.

Completeness. Completeness is defined as a measure of the amount of usable data collected compared to the total amount of data that was expected to be obtained. This will be determined upon final assessment of the analytical results. Three measures of completeness are defined: (1) sampling completeness, defined as the number of valid samples collected relative to the number of samples planned for collection, (2) analytical completeness, defined as the number of valid sample measurements relative to the number of valid samples collected, and (3) overall completeness, defined as the number of valid sample measurements relative to the number of samples planned for collection. Calculation methods for determining completeness are provided in **Section B.4.4**. Overall project completeness is expected to be at least 90 percent.

Sensitivity. Sensitivity refers to the ability of an analytical procedure to quantify an analyte at a given concentration. The sensitivity requirements should be established such that the laboratory reporting limits (RLs) are at or below the relevant and applicable regulatory limits for each contaminant of concern for the project. Method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured, and reported with a 99 percent confidence that the analyte concentration is greater than zero and is determined from repeated analysis of a sample in a given matrix containing the analyte. MDLs have been determined as required in Title 40 of the Code of Federal Regulation Part 136B. The RL is greater than or equal to the lowest standard used to establish the calibration curve. Results greater than the MDL and less than the RL will be qualified estimated (J) by the laboratory. Non-detected results will be reported at the RL. The MDLs and RLs for the constituents of concern are provided in **Tables 1a and 1b**.

A.7. Distribution List

The following individuals will receive copies of the approved QAPP and any subsequent revisions/addenda.

QAPP Distribution List			
Company/ Organization	**Title**	**Name**	**Contact Information**
NYSDEC	Project Manager/Quality Assurance Manager	Jolene Lozewski	518-402-8805 jolene.lozewski@dec.ny.gov
Town of Islip	Project Manager	Robert Schneider	631-467-3315 rschneider@islipny.gov
Arcadis	Principal in Charge/Project Officer	Steve Feldman	Steven.Feldman@arcadis.com 631-391-5244
Arcadis	Project Manager	David Stern	David.Stern@arcadis.com 631-391-5284
Arcadis	Task Manager	Soma Das	Soma.Das@arcadis.com 631-391-5247
Arcadis	Quality Assurance Manager (QAM)	Jennifer Singer	Jennifer.singer@arcadis.com 716-667-6664
Eurofins	Laboratory Project Manager	Julie Gilmore	Julie.Gilmore@et.eurofinsus.com 484-685-0865
SGS	Laboratory Project Manager	Marie Meidhof	Marie.Meidhof@sgs.com 609-752-6106

QAPP Distribution List			
Company/ Organization	**Title**	**Name**	**Contact Information**
Pace	Laboratory Project Manager	Kathy Smith	Kathy.Smith@pacelabs.com 912-465-7901

A.8. Project Organization

On behalf of the Town, Arcadis has overall responsibility for implementing the site characterization investigation described in the RIWP. Arcadis personnel will perform related sampling activities, data evaluation, and preparation of the deliverables as specified in the RI/FS Work Plan. Project direction will be provided by the Town, with oversight by the NYSDEC. A list of key project management personnel is provided below.

SGS in Dayton, New Jersey and Eurofins in Edison, New Jersey will be the subcontracted laboratories for analysis of soil and groundwater samples for PFAS by USEPA 1633 and TCL/TAL parameters. Pace Analytical Services in West Columbia, South Carolina will be the subcontracted laboratory for analysis of PFAS by ASTM D8421.

The management, technical, and QA/QC responsibilities of the key project personnel for implementation of the future sampling activities are summarized as follows:

- NYSDEC Project Manager
  - Provide NYSDEC review and approval of the RI/FS Work Plan, supporting documents, and future deliverables, and
  - Monitor progress of site activities.
- NYSDEC Quality Assurance Manager
  - Review and approval of the QAPP,
  - Review of the quality assurance and quality control portion of any submitted report,
  - Ensure that all activities are performed in compliance with applicable Federal, State, and regional requirements, and
  - Perform field and laboratory audits, if necessary.
- Town of Islip Project Manager
  - Provide overall direction of site actions,
  - Direct Arcadis, and
  - Review Arcadis work products, including data, memoranda, letters, reports, and all other documents transmitted to the NYSDEC.
- Arcadis Principal in Charge/Project Officer
  - Oversee Arcadis work products, and
  - Provide Arcadis approval for major project deliverables.
- Arcadis Project Manager
  - Manage and coordinate the project as defined in the RI/FS Work Plan, with an emphasis on adhering to the objectives of the site activities,

- Perform overall QA/QC of the relevant portions of the site activities,
- Review documents prepared by Arcadis, and
- Ensure that corrective actions are taken for deficiencies cited during any audits of site activities.

- Task Managers

The staff performing the remedial investigation will be directed by various Task Managers. The personnel responsible for specific site activities are listed below.

Task Managers			
Company/ Organization	**Title**	**Name**	**Contact Information**
Arcadis	Project Task Manager	Soma Das	Soma.Das@arcadis.com 631-391-5247
Arcadis	Health and Safety Officer	Kevin Held	Kevin.Held@arcadis.com 908-304-5236
Arcadis	Database Administrator	Kenneth Varley	Kenneth.Varley@arcadis.com 267-685-1842
Arcadis	Data Validator	Lisa Horton	Lisa.Horton@arcadis.com 631-391-5222

Duties of each Task Manager include, as appropriate:

- Manage relevant day-to-day activities,
- Develop, establish, and maintain files on relevant site activities,
- Review data reduction from the relevant site activities,
- Perform final data review of field data reduction and reports on relevant site activities,
- Ensure corrective actions are taken for deficiencies cited during audits of relevant site activities,
- Review relevant field records and logs,
- Instruct personnel working on relevant site activities,
- Coordinate field and laboratory schedules pertaining to relevant site activities,
- Request sample bottles from laboratory,
- Review field instrumentation, maintenance, and calibration to meet quality objectives,
- Ensure that field activities are performed in accordance to the QAPP,
- Prepare reports pertaining to relevant site activities, and
- Maintain field and laboratory files of logbooks/logs, data reductions, and calculations and transmit originals to the Project Manager.

- Arcadis Field Personnel
  - Perform field procedures as set forth in the RI/FS Work Plan and FSP and collect quality assurance samples,
  - Perform field oversight of subcontractors,
  - Calibrate, operate, and maintain field equipment,
  - Maintain sample custody,
  - Prepare field records and logs,
  - Reduce field data, and
  - Prepare draft reports, data tables and graphs.

- Contractor Quality Assurance Manager
  - Oversee planning, documenting, coordination of the QAPP,
  - Assess effectiveness of the QAPP, including QA audits,
  - Review and discuss quality-related issues, if identified,
  - Verify deficiencies, if identified, are corrected,
  - Review laboratory data corrective action, as necessary for QA compliance,
  - Perform or coordinate analytical data validation and review,
  - Review laboratory QA/QC,
  - Review data validation and review documentation,
  - Initiate corrective action, as necessary, for QA compliance, and
  - Liaison between the assigned laboratory and the contractor
- Laboratory Project Manager(s)
  - Ensure resources are available on an as-required basis,
  - Coordinate analyses and chain of custody,
  - Oversee review of data,
  - Direct implementation of corrective actions required as a result of data review, internal audits, or external audits (if required),
  - Oversee preparation of analytical reports, and
  - Approve final analytical reports and case narratives prior to submission to the Contractor.
- Analytical Laboratory QA/QC Officer
  - Review laboratory QA and QC,
  - Review QA/QC documentation,
  - Investigate project-related nonconformances, and
  - Verify resolution of such nonconformances.

A.9. Project Quality Assurance Manager Independence

The Project QAM will be independent of environmental information operations team (i.e. the end users of the data). The QAM will not have a functional role in the collection or analysis of the field investigation data or in the operations on budgeting of the project, thus ensuring independence of the QAM evaluation of data quality and implementation of the QAPP. The QAM is also not involved in the preparation of reports to the client, agencies, and other stakeholders.

A.10. Project Organizational Chart and Communications

The Organization Chart is provided as Appendix G of the RI/FS Work Plan.

A.11. Personnel Training/Certification

In compliance with the Occupational Safety and Health Administration (OSHA) final rule, “Hazardous Waste: Operations and Emergency Response [29 Code of Federal Regulations 1910.120(e)]”, all personnel performing sampling activities at the site will have completed the requirements for OSHA 40-Hour Hazardous Waste Operations and Emergency Response training. Persons in field supervisory positions will have also completed the additional OSHA 8 Hour Supervisory Training.

Field personnel involved with the shipping of samples to the subcontracted laboratory will have completed the United States Department of Transportation (USDOT) Hazardous Materials (HazMat) #1 – USDOT/International Air Transportation Association HazMat Shipping and Transportation.

Arcadis’ Human Resources Department maintains records to document management and technical training accomplished by each staff member. Training documentation records are stored in the Arcadis Training Center database. As appropriate and available, training to meet the OSHA and USDOT specifications will be formal in-person or on-line training. The project manager will ensure that field crews have or receive training on the required sampling activities prior to mobilization to the site to ensure that work is conducted correctly and safely. Arcadis will track the training for Arcadis employees and subcontractors, and all training expirations will be communicated to the appropriate staff and supervisors.

The subcontracted laboratory performing analytical work must have the knowledge and skills in execution of the analytical methods being requested. Information on laboratory staff competence is provided in the laboratory Quality Assurance Plan. The laboratories are certified by the New York State Department of Health (NYSDOH).

A.12. Documents and Records

A.12.1. General

Samples will be collected as described in the FSP. Detailed descriptions of the documentation and reporting requirements are presented below.

A.12.2. Sample Designation System

Samples will be identified with a unique designation system that will facilitate sample tracking. The sample designation system to be employed during the sampling activities will be consistent, yet flexible enough to accommodate unforeseen sampling events and conditions. An alpha-numeric system is considered appropriate and will be used by field personnel to assign each sample with a unique sample identification number. The proposed sample IDs and sampling intervals for soil and groundwater are included in Tables 2 and 3 of the RI/FS Work Plan.

Sample identifications will follow the following format for consistency with the nomenclature assigned to samples:

- Parent soil samples: Site Abbreviation-Area #-SO-Boring #(Upper Depth-Lower Depth)-Sample Collection Date

  Example: ISP-1-SO-2(34-36)-07312024

- New and existing monitoring well parent groundwater samples: MW-Area #-Well #-Sample Collection Date

  Example: MW-1-1-07312024

- Parent groundwater samples: Site Abbreviation-Area #-GW-Sample #(Upper Depth-Lower Depth)-Sample Collection Date

  Example: ISP-4-GW-2(60-63)-07312024

- Field Duplicate: Site Abbreviation-Media Type-FD-Duplicate #-Sample Collection Date

  Example: ISP-SO-FD-2-07312024

- Blank QC samples: Site Abbreviation-QC Sample Abbreviation (e.g., Field Blank [FB], equipment blank [EB], etc.)-QC Sample Type #-Sample Collection Date

  Example: ISP-FB-3-07312024

A.12.3. Field Documentation

Field personnel will provide comprehensive documentation covering all aspects of field sampling, field analysis, and sample chain of custody. This documentation constitutes of a record that allows reconstruction of all field events to aid in the data review and interpretation process. All documents, records, and information relating to the performance of the field work will be retained in the project file.

The various forms of documentation to be maintained throughout the action include:

Daily Production Documentation – A field logbook consisting of a waterproof, bound notebook that will contain a record of all activities performed at the site. Alternatively, field notes may be maintained in an electronic format.

Sampling Information – Detailed notes will be made as to the exact sampling location, physical observations, and weather conditions (as appropriate).

Sample Chain of Custody – Chain of custody forms will provide the record of responsibility for sample collection, transport, and submittal to the laboratory. Chain of custody forms will be filled out at each sampling site, at a group of sampling sites, or at the end of each day of sampling by Arcadis field personnel designated to be responsible for sample custody. In the event the samples are relinquished by the designated sampling person to other sampling or field personnel, the chain of custody form will be signed and dated by the appropriate personnel to document the sample transfer. The original chain of custody form will accompany the samples to the laboratory, and copies will be forwarded to the project files.

Field Equipment, Calibration, and Maintenance Logs – To document the calibration and maintenance of field instrumentation, calibration and maintenance logs will be maintained for each piece of field equipment that is not factory-calibrated. Alternatively, a description of the calibration may be included in the field notebook.

A.12.4. Laboratory Documentation Files

A.12.4.1 Laboratory Project Files

The laboratory will establish a file for all pertinent data. The file will include all correspondence, email, phone logs, and chain of custody forms. The laboratory will retain all project files and data packages for a period of five years.

A.12.4.2 Laboratory Logbooks

Workbooks, bench sheets, instrument logbooks, and instrument printouts will be used to trace the history of samples through the analytical process and document important aspects of the work, including the associated quality controls. As such, logbooks, bench sheets, instrument logs, and instrument printouts will be part of the permanent record of the laboratory.

Each page or entry will be dated and initialed by the analyst at the time of entry. Errors in entry will be crossed out in indelible ink with a single stroke, corrected without the use of white-out or by obliterating or writing directly over the erroneous entry, and initialed and dated by the individual making the correction. Pages of logbooks that are not used will be completed by lining out unused portions.

Information regarding the sample, analytical procedures performed, and the results of the testing will be recorded on laboratory forms or personal notebook pages by the analyst. These notes will be dated and will also identify the analyst, the instrument used, and the instrument conditions.

Laboratory notebooks will be periodically reviewed by the laboratory group leaders for accuracy, completeness, and compliance to this QAPP. All entries and calculations will be verified by the laboratory group leader. If all entries on the pages are correct, then the laboratory group leader will initial and date the pages. Corrective action will be taken for incorrect entries before the laboratory group leader signs.

A.12.4.3 Computer Tape and Hard Copy Storage

All electronic files and deliverables will be retained by the laboratory for a minimum of five years; digital data packages will also be retained for a minimum of five years.

A.12.5. Data Reporting Requirements

Data will be reported both in the field and by the analytical laboratory, as described below.

A.12.5.1 Field Data Reporting

Information collected in the field through visual observation, manual measurement, and/or field instrumentation will be recorded in field digital or paper logbooks or data sheets and/or on forms. Such data will be reviewed by the appropriate task leader for adherence to this QAPP and for consistency. Concerns identified because of this review will be discussed with the field personnel, corrected if possible, and, as necessary, incorporated into the data evaluation process.

If applicable, field data forms and calculations will be processed and included in appendices to the appropriate reports (when generated). The original field logs, documents, and data reductions will be kept in the project file at the Arcadis office in Melville, New York.

A.12.5.2 Laboratory Data Reporting

Analytical results will be provided by the laboratory in both a digital and a hard copy format. Sample results will be provided in a standard 2-week turnaround time (unless otherwise specified).

Laboratory reports will be provided in a format consistent with the NYSDEC Analytical Services Protocol (ASP) Category B reporting requirements. The laboratory report will be provided in digital (i.e., PDF) format. Category B laboratory reports include the following items, dependent upon the requirements of the analytical method:

- Chain of custody
- Case narrative
- Final parameter concentration for all samples
- Preparation or extraction and analysis dates/times
- Calibration standards
- Surrogate recoveries

- Blank results
- Spike recoveries
- Duplicate results
- Confirmation samples
- Internal standard area and retention time summary
- Chromatograms
- Raw data files for all field samples and associated QA/QC samples
- Sample preparation worksheets and instrument run logs
- Other specific information as required by the analytical method

Analytical data will be provided in NYSDEC-compliant electronic data deliverable (EDD) format and will be imported in the project EQuIS database. The EDD will be provided in the EQuIS EFWEDD four-file format (Sample, Test, Result, Batch).

The data packages and EDDs will be examined to ensure that the correct analyses were performed for each sample submitted and that all of the analyses requested on the chain of custody form were performed. If discrepancies are noted, the QAM will be notified and will promptly follow up with the laboratory to resolve any issues.

A.12.6. Project File

All records generated during this project will be kept on file by Arcadis on behalf of the Town of Islip. These records may include field logbooks, field sampling forms, chain of custody forms, laboratory data deliverables, photographs, and other relevant records. To the extent practicable, records will be stored electronically. Arcadis maintains project files at its Melville, New York office. Record retention shall be in accordance with client- and project-specific requirements and Arcadis policies, the most stringent of which will apply.

B. Group B: Implementing Environmental Information Operation

B.1. Identification of Project Environmental Operations

As described in the RI/FS Work Plan and in **Section A.5** of this QAPP, the project environmental operations include drilling and sampling of soil from soil boring; drilling and sampling of soil and groundwater from VAPs; drilling, installing, and development of new permanent monitoring wells; and collecting groundwater samples from new and existing monitoring wells. A total of 35 soil borings are proposed in the RI/FS Work Plan, with provisional borings to be drilled/samples as needed based on the analytical results of the initial sampling. A total of 21 VAPs are proposed to be drilled at depths ranging from 150 to 300 feet below land surface. Up to 15 new groundwater monitoring wells are proposed to be installed as part of the RI/FS.

The data generated by the project environmental operations will be used to characterize the nature and extent of PFAS contamination in soil and groundwater on site. Data quality objectives will be met through implementation of the procedures outlined below for field and laboratory operations. The field operation methods have been developed, tested, reviewed, and refined by experience field technicians and have found to produce data of

acceptable quality when implemented at other sites. Laboratory operation methods have been demonstrated to meet method standards as applicable and are monitored as described in **Section B.4**. Additionally, the field and laboratory operation methods presented below are measured using the performance indicators described in **Section A.6.2** and evaluated through the QA/QC process with oversight by the QAM.

B.2. Methods for Environmental Information Acquisition

This section describes the protocols that will be followed for soil sample collection, groundwater sample collection, and laboratory analyses. QA/QC activities and procedures are presented in this QAPP and attached standard operating procedures (SOPs; **Appendices A and B**). The Arcadis task leader overseeing field sampling operations for the project will also be responsible for maintaining and updating the SOPs as necessary.

Sampling locations have been selected based on consideration of previous sampling results. The sampling will be focused on nine Areas, defined above in **Section A.6.1.4**. In addition, several "facility-wide" locations have been proposed for VAP borings and groundwater sample collection. The proposed scope of work and associated rationale for each Area and "facility-wide" activity is provided in Table 1 of the RI/FS Work Plan.

B.2.1. Sampling Methods

Field activities will be performed in accordance with Arcadis Quality Procedures (QP) and TGIs (copies included in **Appendix A**).

General procedures include:

- Field Activities Documentation, Arcadis QP 3.06, Revision 2, July 19, 2023
- Groundwater and Soil Sampling Equipment Decontamination, Arcadis TGI, Revision 3, August 30, 2023
- Investigation-Derived Waste Handling and Storage, Arcadis TGI, Revision 1, May 15, 2020.
- Resource Conservation and Recovery Act Waste Characterization Sampling, Arcadis TGI, Revision 0, May 20, 2021.
- Sample Chain of Custody, Arcadis TGI, Revision 3, March 28, 2022.

Applicable procedures for groundwater will include:

- In-Situ and Ex-Situ Water Quality Parameters – Surface Water and Groundwater, Arcadis TGI, Revision 1, April 15, 2022.
- Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells, Arcadis TGI, Revision 3, April 5, 2023.
- Manual Water-Level and NAPL Monitoring, Arcadis TGI, Revision 5, March 8, 2024.
- Monitoring Well Development, Arcadis TGI, Revision 2, April 5, 2023.
- Monitoring Well Inspection Assessment, Arcadis TGI, Revision 1, June 30, 2022.
- Monitoring Well Installation, Arcadis TGI, Revision 1, June 23, 2022.
- Vertical Aquifer Profile Sampling, Arcadis TGI, Revision 2, June 15, 2022.

Applicable procedures for groundwater will include:

- Extraction/Preservation of Soil/Sediment for VOCs, Revision 2, May 11, 2020.
- Soil Description, Arcadis TGI, Revision 4, June 14, 2022.

- Soil Drilling and Sample Collection, Arcadis TGI, Revision 3, April 5, 2023.
- Utility Locating Using Radio Frequency Methods, Arcadis TGI, Revision 4, February 3, 2022

Applicable procedures for PFAS sampling of soil and groundwater will include:

- Equipment and Reagent Blank Sample Collection for PFAS Analysis, Arcadis TGI, Revision 2, February 21, 2022.
- PFAS Field Sampling Guide, Arcadis TGI, Revision 12, September 20, 2023.
- Sampling, Analysis, and Assessment of PFAS Under NYSDEC's Part 375 Remedial Programs, NYSDEC, April 2023.

B.2.3. Equipment Decontamination

Field personnel will follow the decontamination procedures outlined in the Arcadis TGI, Groundwater and Soil Sampling Equipment Decontamination (copy included in **Appendix A**). Non-disposable equipment will be cleaned between use for each sample location.

B.2.4. Laboratory Analyses and Analytical Method Requirements

Samples in accordance with relevant analytical methods and laboratory-specific SOPs. SGS in Dayton, New Jersey and Eurofins in Edison, New Jersey will be the subcontracted laboratories for analysis of soil and groundwater samples for PFAS by USEPA 1633 and TCL/TAL parameters. Pace Analytical Services in West Columbia, South Carolina will be the subcontracted laboratory for analysis of PFAS by ASTM D8421. Copies of laboratory SOPs are included in **Appendix B**. Analyses will be performed following the quality control limits listed in **Table 2**. Results will be compared to the NYSDEC 6 NYCRR Part 375 Restricted Use Soil Cleanup Objectives, NSDEC Division or Water Technical and Operational Guidance Series Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations, and NYSDEC Guidance Values for PFAS (**Table 2**).

The methods that pertain to groundwater and soil sampling activities include:

- PFAS by ASTM D8421 and USEPA 1633
- TCL VOCs by USEPA method 8260D
- TCL SVOCs including 1,4-dioxane by USEPA method 8270E and 8270E-SIM
- TCL PCBs by USEPA method 8082B
- TCL pesticides by USEPA method 8081B
- TCLP herbicides by USEPA method 8151A
- TAL metals by USEPA method 6010C, 7470A, and 7471B
- Hexavalent chromium (USEPA method 7196A)

B.2.5. Existing Information

Arcadis prepared the Records Search Report (RSR) as part of the site characterization (SC) process. Based on the findings of the RSR/SC, NYSDEC required that an RI/FS be conducted at the Site. The existing Site data was utilized to support the proposed sampling locations.

B.3. Integrity of Environmental Information

The procedures for ensuring the integrity of the environmental information operations are described in this Section and include procedures and methods for handling and custody of field samples as well as samples submitted for laboratory analyses.

The integrity of field samples is ensured through the use of proper sample collection, packaging, and transport methods, maintaining and recording chain of custody, and documenting appropriate preservation and holding conditions.

Once submitted to the laboratory for analysis, sample integrity is ensured through the use of NYSDOH certified laboratories. Laboratory accreditation will be confirmed by the QAM prior to sample submittal and annually thereafter, if necessary. As noted in **Section B.2.4**, SGS, Eurofins, and Pace are the subcontracted laboratories for analysis of groundwater and soil samples. The laboratories are currently NYSDOH certified. In the event that either laboratory is unable to perform the necessary analyses, alternate laboratories in their networks are available.

B.3.1. Sample Containers and Preservation

Appropriate sample containers, preservation methods, and laboratory holding times are shown in **Table 4**.

The analytical laboratory will supply appropriate sample containers and preservatives, as necessary. The bottles will be purchased by the laboratory, pre-cleaned according to USEPA Office of Solid Waste and Emergency Response Directive 9240.05A requirements (USEPA 1992b). The field personnel will be responsible for properly labeling containers and preserving samples.

B.3.2. Field Custody Procedures

The objective of field sample custody is to assure that samples are not tampered with from the time of sample collection through time of transport to the analytical laboratory. Persons will have “custody of samples” when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

Field custody documentation consists of both field logbooks and field chain of custody forms. An example of chain of custody form is provided in Arcadis TGI, Sample Chain of Custody (copy included in **Appendix A**).

B.3.2.1. Field Logbooks

Field logbooks will provide the means of recording data collecting activities performed. As such, entries will be described in as much detail as possible so that persons going to the site could reconstruct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or logbooks. Logbooks will be assigned to field personnel but will be stored in a secure location when not in use.

The title page of each logbook will contain the following:

- Project name;
- Project start date; and

- End date.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling team personnel, and the purpose of their visit will also be recorded in the field logbook. To ensure at any future date that logbook pages are not missing, each page will be sequentially numbered. Erroneous entries will be corrected by crossing out the original entry, initialing it, and then documenting the proper information.

Measurements made and samples collected will be recorded either in the field logbook or on sampling forms. Entries will be made in ink, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected or a measurement is made, a detailed description of the location shall be recorded. The numbers of the photographs taken of the location, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration, if relevant. In addition, certain media sampling locations will be surveyed to accurately record their locations. If different than the field crew, the survey crew will use their own field logs and will supply the sampling location coordinates to the Project Coordinator who will ensure they are incorporated into the project database.

The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume, and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

B.3.2.2. Sample Labelling

As appropriate, preprinted sample labels will be affixed to sample bottles prior to delivery at the sampling site or sample bottles will be labeled at the site during sample collection. The following information is required on each sample label:

- sample media;
- project number and site name;
- sample identification code and other sample identification information, if applicable;
- analysis required;
- date collected;
- time sampled;
- initials of sampling personnel;
- sample type (e.g., composite or grab); and
- preservative added, if applicable.

B.3.2.3. Field Chain of Custody Forms

Completed chain of custody forms will be required for all samples to be analyzed. Chain of custody forms will be initiated by the sampling crew in the field. The chain of custody forms will contain the unique sample identification number, sample date and time, sample media, sample type, preservation (if any), and analyses required. The original chain of custody form will accompany the samples to the laboratory. Copies of the chain of custody will be

made prior to shipment (or multiple copy forms used) for field documentation. The chain of custody forms will remain with the samples at all times. The samples and signed chain of custody forms will remain in the possession of the sampling crew until the samples are delivered to the express carrier (e.g., FedEx) or hand delivered to a mobile or permanent laboratory or placed in secure storage.

Whenever samples are split with a government agency or other party, a separate chain of custody will be prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representatives' signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.

B.3.3. Management of Investigation Derived Materials and Wastes

Management of investigation-derived materials and wastes will be performed consistent with the USEPA guidance Guide to Management of Investigation-Derived Wastes (USEPA 1992a). Disposable equipment (including personal protective equipment) and debris will be containerized and appropriately labeled during the sampling events and will be disposed of accordingly. All water generated during equipment decontamination will be containerized and temporally staged onsite in a USDOT approved 55-gallon drum and will be disposed of appropriately based on analytical results or generator knowledge.

B.3.4. Chain of Custody, Handling, Packing, and Shipping Requirements

All shipping or sample transfer information will be recorded at the end of each day or collection period on chain of custody forms. The field sampler is responsible for custody of the samples until they are delivered to the laboratory or picked up for shipping. (Note: As few people as possible will handle the samples to ensure sample custody.) Each time one person relinquishes control of the samples to another person, both individuals must complete the appropriate portions of the chain-of-custody form by filling in their signature as well as the appropriate date and time of the custody transfer. During transport by a commercial carrier, the air bill will serve as the associated chain-of-custody. Once at the laboratory, the sample receipt coordinator will open the coolers and sign and date the chain-of-custody form. The laboratory personnel are then responsible for the care and custody of samples (see **Section B.3.5**).

A sample is considered to be in one's custody if:

- The sample is in the sampler's physical possession,
- The sample has been in the sampler's physical possession and is within sight of the sampler,
- The sample is in a designated, secure area, and/or
- The sample has been in the sampler's physical possession and is locked up.

Sample packaging and shipment procedures are designed so that the samples will arrive at the laboratory with the chain of custody intact.

It is imperative that all samples are submitted to the laboratory with ample time for the analysis to be completed within the method-specified holding time. It should be noted that the laboratory needs to be aware that missing a holding time is unacceptable and may result in unusable data if the holding time is missed.

General sample packaging and shipping requirements are as follows:

- Securely affix the sample label to the container with clear packing tape.
- Check the cap on the sample container to confirm that it is properly sealed.

- Complete the chain of custody form with the required sampling information and confirm that the recorded information matches the sample labels. NOTE: If the designated sampler relinquishes the samples to other sampling or field personnel for packing or other purposes, the sampler will complete the chain of custody prior to this transfer. The appropriate personnel will sign and date the chain of custody form to document the sample custody transfer.
- Using duct tape, secure the outside drain plug at the bottom of the cooler.
- Wrap glass sample containers in bubble wrap or other cushioning material.
- Place 1 to 2 inches of cushioning material at the bottom of the cooler.
- Place the sealed sample containers into the cooler.
- Place ice in plastic bags, seal the bags, and place the bags loosely in the cooler.
- Fill the remaining space in the cooler with cushioning material.
- Place chain of custody forms in a plastic bag and seal. Tape the forms to the inside of the cooler lid.
- Close the lid of the cooler and secure with duct tape.
- Wrap strapping tape (or equivalent) around both ends of the cooler at least twice.
- Mark the cooler on the outside with the shipping address and return address, affix “Fragile” labels, and draw (or affix) arrows indicating “this side up.” Cover the labels with clear plastic tape. Place a signed custody seal over the cooler lid. NOTE: If the samples are being delivered directly to the laboratory or will be picked up by the lab’s courier service, this step is eliminated.

Samples will be packaged by field personnel and transported as low-concentration environmental samples. The samples will be hand-delivered or delivered by an express carrier within 48 hours of the time of collection. In some cases, the analytical method may require analysis within a shorter holding time, and arrangements will need to be made to accommodate the laboratory requirements. Shipments will be accompanied by the chain of custody form identifying the contents. The original form will accompany the shipment; copies will be retained by the sampler for the office records. If the samples are sent by common carrier, a bill of lading will be used. Receipts or bills of lading will be retained as part of the permanent project documentation. Commercial carriers are not required to sign off on the chain of custody form as long as the forms are sealed inside the sample cooler, and the custody seals remain intact.

Custody seals and cooler packing materials will be provided by the analytical laboratory. The filled, labeled, and sealed containers will be placed in a cooler on ice and carefully packed to eliminate the possibility of container breakage.

B.3.5. Laboratory Custody Procedures

B.3.5.1. General

Upon sample receipt, laboratory personnel will be responsible for sample custody. The original field chain of custody form will accompany all samples requiring laboratory analysis. The laboratory will use chain of custody guidelines described in the USEPA guidance documents. Samples will be kept secured in the laboratory until all stages of analysis are complete. All laboratory personnel having samples in their custody will be responsible for documenting and maintaining sample integrity.

B.3.5.2. Sample Receipt and Storage

Immediately upon sample receipt, the laboratory sample custodian will verify the custody seal (if present), open the cooler, compare the contents against the field chain of custody, and check sample preservation as appropriate. If a sample container is missing, a sample container is received broken, the sample is in an inappropriate container, or has not been preserved by appropriate means, Arcadis will be notified. The laboratory sample custodian will be responsible for logging the samples in, assigning a unique laboratory identification number to each sample, labeling the sample bottle with the laboratory identification number, and moving the sample to an appropriate storage location to await analysis. The project name, date sampled, date received, matrix, analysis required, storage location and date, will be recorded in the laboratory tracking system. Relevant custody documentation will be placed in the project file.

B.3.5.3. Sample Analysis

Analysis of an acceptable sample will be initiated by worksheets that contain all pertinent information for analysis.

Samples will be organized into sample delivery groups (SDGs) by the laboratory. An SDG may contain up to 20 field samples (field duplicates and rinse blanks are considered field samples for the purposes of SDG assignment). All field samples assigned to a single SDG shall be received by the laboratory within a maximum of seven calendar days and must be processed through the laboratory (preparation, analysis, and reporting) as a group. Every SDG must include a minimum of one site-specific MS/MSD pair, which shall be received by the laboratory at the start of the SDG assignment.

B.3.5.4. Sample Storage Following Analysis

Samples will be maintained by the laboratory for at least 30 days after the final report is delivered to Arcadis. The laboratory will be responsible for the eventual and appropriate disposal of the samples. Unused portions of the samples, sample extracts and associated wastes will be disposed of by the laboratory in accordance with applicable rules and regulations as specified in their SOP for waste disposal.

B.4. Quality Control

This section details the QA/QC measures that will be implemented during the 2024 investigation to minimize variability, mitigate the potential for false positive and/or false negative error, and increase accuracy and defensibility in the collected data.

B.4.1. Laboratory Quality Assurance/Quality Control

The subcontracted laboratories are NYSDOH certified for the requested analyses. Laboratories are routinely audited as part of the laboratory certification process. The laboratory will notify the Arcadis Project Manager if any audit findings or proficiency testing failures may affect the reported results. Laboratory SOPs and certifications are included in **Appendix B**.

Laboratory QA/QC steps and QC sample analyses will be consistent with USEPA requirements and industry standards. In addition, performance evaluation samples may be included to evaluate laboratory analytical performance.

Internal laboratory quality control checks will be used to monitor data integrity. These checks will include method blanks, MS/MSDs, LCS, labeled compounds/internal standards, and calibration standards. Project quality control limits are identified in **Table 2**.

B.4.1.1. Method Blanks

Sources of contamination in the analytical process, whether specific analyses or interferences, need to be identified, isolated, and corrected. The method blank is useful in identifying possible sources of contamination within the analytical process. For this reason, it is necessary that the method blank is initiated at the beginning of the analytical process and encompasses all aspects of the analytical work. As such, the method blank would assist in accounting for any potential contamination attributable to glassware, reagents, instrumentation, or other sources which could affect sample analysis. One method blank will be analyzed with each analytical series associated with no more than 20 samples.

B.4.1.2. Matrix Spikes/Matrix Spike Duplicates

An MS is an aliquot of an environmental samples to which known quantities of target analytes are added in the laboratory. An MSD is a second aliquot of the samples that is also spiked with identical concentrations of target analytes. The MS and MSD are analyzed in a manner identical to that of the sample. The purpose of an MS is to determine the accuracy of the overall analytical procedure for determining the analytes of concern in the sample. The MSD analysis is used to document both the precision and accuracy of the method in each sample matrix.

To get the true benefit from MS and MSD analyses, it is necessary that the sample come from the project Site so that it is possible to ascertain if the matrix itself is contributing to analytical difficulties or outliers. As required by the analytical method, MS/MSD pairs will be analyzed at a five percent frequency as shown in **Table 3** (i.e., additional sample volume for MS and MSD analysis will be collected for every twenty field samples collected per matrix). When MS recoveries are outside quality control limits, associated LCS and surrogate spike recoveries will be evaluated (as applicable) to attempt to verify the reason for the deviation and determine the effect on the reported sample results.

B.4.1.3. Laboratory Control Samples

An LCS is a blank matrix, free from the analytes of interest, spiked with verified known amounts of analytes created from a source other than that used to make up calibration standards. PFAS analysis also includes a low-level LCS (LLLCS). The LCS and LLLCS is carried through the analysis along with the samples. The intent of LCS and LLLCS analysis is to provide insight into the intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system. This includes the preparation of calibration standards, validity of calibration, sample preparation, instrument setup, and the premises inherent in quantitation. One LCS and LLLCS will be analyzed with each analytical series associated with not more than twenty samples.

B.4.1.4. Surrogate Compounds

A surrogate spike is an organic non-target analyte that is unlikely to occur under natural conditions but that has chemical properties similar to those of the analytes of interest. The surrogate standard is added to the sample in a known amount before purging or extraction and is used to evaluate the response of the analyte to the preparation and analysis procedures. The surrogate concentration is measured using the same procedures used to measure other analytes in the sample. The surrogate spike is used to provide broader insight into the proficiency and efficiency of an analytical method on a sample-specific basis. This control reflects analytical conditions that may not be attributable to sample matrix.

If surrogate spike recoveries exceed specified QC limits, the analytical results must be evaluated thoroughly in conjunction with other control measures. In the absence of other control measures, the integrity of the data may not be verifiable, and reanalysis of the samples with additional control may be necessary.

Surrogate spike compounds will be selected using the guidance provided in the analytical methods and will be added to all field samples and laboratory quality control samples.

B.4.1.5. Isotopically Labeled Compounds

Isotopically labeled compounds are an analog of a target analyte which have been synthesized with one or more atoms in the structure replaced by a stable (non-radioactive) isotope of that atom and include carbon-13 or deuterium. These labeled compounds are used to isotope dilution quantification. PFAS analysis uses these isotopically labeled compounds are used as both extracted internal standards (EIS) and non-extracted internal standards (NIS) in this method.

EIS compounds are added to every standard, blank, sample, and quality control sample at the beginning of the sample preparation process are any used to quantify the native target analyte concentration. If EIS areas exceed the specified tolerances, the cause will be investigated, and the samples may be reanalyzed.

NIS are labelled PFAS compounds spiked into the concentrated extract immediately prior to injection of an aliquot of the extract into the instrument. NIS compounds are added to every standard, blank, sample, and quality control sample. If NIS responses are not within limits, the cause will be investigated, and the sample will be reanalyzed.

The acceptability of isotopically labelled compound recoveries will be determined using the guidance provided within the analytical methods.

B.4.1.6. Calibration Check Standards

Calibration check standards analyzed within a particular analytical series provide insight regarding the instruments' stability. A calibration check standard is analyzed at frequency specified in the applicable method and/or the lab's SOP.

In general, calibration check standards will be analyzed after every 12 hours, or more frequently, as specified in the applicable analytical method. In analyses where internal standards are used, a calibration check standard will only be analyzed in the beginning of an analytical series. If results of the calibration check standard exceed specified tolerances, then all samples analyzed since the last acceptable calibration check standard will be reanalyzed. Laboratory instrument calibration standards will be selected utilizing the guidance provided in the analytical methods.

B.4.2. Data Precision Assessment Procedures

Field precision is difficult to measure because of temporal variations in field parameters. However, precision will be controlled through the use of experienced field personnel, properly calibrated meters, and duplicate field measurements. Field duplicate samples will be used to assess precision for the entire measurement system including sampling, handling, shipping, storage, preparation, and analysis.

Laboratory data precision for analyses will be monitored through the use of MS/MSD and laboratory duplicates.

The precision of data will be measured by calculation of the RPD by the following equation:

$$RPD = \frac{(A - B)}{(A + B)/2}\ x\ 100$$

Where:

A = Analytical result from one of two duplicate measurements

B = Analytical result from the second measurement

Precision objectives for field duplicate, MSD, and laboratory duplicate analyses are identified in **Table 2**.

B.4.3. Data Accuracy Assessment Procedures

The accuracy of field measurements will be controlled by experienced field personnel, properly calibrated field meters, and adherence to established protocols. The accuracy of field meters will be assessed by review of calibration and maintenance logs.

Laboratory accuracy will be assessed via the use of MS, LCS, LLLCS, surrogate compounds, EIS, and NIS. Accuracy will be calculated in terms of percent recovery as follows:

$$\%Recovery = \frac{A - X}{B} \; x \; 100$$

Where:

A = Value measured in spiked sample or standard

X = Value measured in original sample

B = True value of amount added to sample or true value of standard

This formula is derived under the assumption of constant accuracy over the original and spiked measurements. If any accuracy calculated by this formula is outside of the acceptable levels, data will be evaluated to determine whether the deviation represents unacceptable accuracy, or variable, but acceptable accuracy. Accuracy objectives for MS, LCS, LLLCS, surrogate compounds, EIS, and NIS are identified in **Table 2**.

B.4.4. Data Completeness Assessment Procedures

Completeness of a field or laboratory dataset will be calculated by comparing the number of collected samples to the planned sample collection and valid sample results generated to the total number of results generated.

$$Field\ Completeness = \frac{Number\ of\ collected\ samples}{Total\ number\ of\ results\ generated} \; x \; 100$$

$$Analytical\ Completeness = \frac{Number\ of\ valid\ results}{Total\ number\ of\ results\ generated} \; x \; 100$$

As a general guideline, overall project completeness is expected to be at least 90 percent. The assessment of completeness will require professional judgment to determine data usability for the intended purposes.

B.4.5. Field Quality Assurance/Quality Control

Field precision will be controlled using experienced field personnel and duplicate field measurements. Field duplicates will be used to assess precision for the entire measurement system including sampling, handling, shipping, storage, preparation, and analysis. Equipment blanks will be used to assess cleanliness of the sampling equipment and effectiveness of cleaning procedures.

Additionally, the following sampling and custody procedures will be used to maintain data quality:

- Accurate and complete labeling of sample containers;

- Correct sample preservation;
- Collection of correct sample volumes for the respective analytical method;
- Accurate and complete chain-of-custody forms;
- Maintenance of label integrity and protection of sample containers against breakage during transport from field to laboratory; and
- Expedition of sample delivery in response to holding time requirements.

B.4.5.1. Field Duplicates

Field duplicates for soil will be collected to verify the reproducibility of the sampling methods. Field duplicates will be prepared by placing well homogenized aliquots from the same sample location into individual sample containers, which are submitted blind to the laboratory. Field duplicates will be analyzed at a 5 percent frequency (every 20 samples) for soil and groundwater as shown in **Table 3**.

B.4.5.2. Equipment Blanks

Equipment blanks will be used to monitor the cleanliness of the soil and groundwater sampling equipment and the effectiveness of the cleaning procedures. One equipment blank per day per crew will be collected from the sampling equipment used for soil and groundwater sampling as shown in **Table 3**. Equipment blanks will be prepared by pouring analyte-free water (supplied by the laboratory) over reusable equipment following the final decontamination process for the day.

If target analytes are found in the equipment blanks, sampling and handling procedures will be reevaluated and corrective actions taken. These may consist of, but are not limited to, obtaining sampling containers from new sources, training of personnel, discussions with the laboratory, invalidation of results, greater attention to detail during the next sampling event, or other procedures felt appropriate.

B.4.5.3. Trip Blank

A trip blank will contain laboratory supplied analyte free water and will be transported to the site and returned to the laboratory without opening. This will serve as a check from contamination originating from sample transport, shipping, and from site conditions. One trip blank per data per cooler will be submitted to the laboratory with the collection of soil and groundwater samples as shown in **Table 3**. The trip blank sample will be submitted for VOC analysis.

B.4.5.4. Matrix Spike/Matrix Spike Duplicates

As noted above in **Section B.4.1.2**, MS/MSDs for soil samples will be used to measure the accuracy of analyte recovery from the sample matrices and will be site-specific. MS/MSD pairs will be analyzed at a 5 percent frequency (every 20 samples) for soil and groundwater as shown in **Table 3**. Additional sample volume will be collected and submitted to the laboratory for site-specific MS/MSD analysis and will be noted on the chain of custody.

B.5. Instrument/Equipment Calibration, Testing, Inspection, and Maintenance

This section details the instruments and equipment that will be used during implementation of the RIWP as well as the procedures and documentation activities that will be performed to ensure that the instruments/equipment are available and in working order when needed.

B.5.1. Field Instruments and Equipment

Calibration checks of field equipment will be performed daily when used. Field equipment operation, calibration, and maintenance procedures are provided in the equipment operation manuals. Field personnel will be required to adhere to the Arcadis TGI In-Situ and Ex-Situ Water Quality Parameters – Surface Water and Groundwater (copy included in **Appendix A**).

Prior to any field sampling, each piece of field equipment will be inspected to confirm that it is operational. If the equipment is not operational, it must be serviced prior to use. All meters that require charging or batteries will be fully charged or have fresh batteries. If instrument servicing is required, it is the responsibility of the Field Sampling Manager to follow the maintenance schedule and arrange for prompt service.

Instrument	Device Type	Instrument Serial No.	Calibration Protocol
Multi-Meter	Multi-parameter sonde with probe-based sensor (pH, Oxidation-Reduction Potential, Dissolved Oxygen, etc.)	Rental unit – Serial will be recorded in Field Notebook	Calibrated according to manufacturer recommendations
Turbidity Meter	Multi-parameter sonde with probe-based sensor (set for turbidity)	Rental unit – Serial will be recorded in Field Notebook	Calibrated according to manufacturer recommendations
Peristaltic Pump	Solinst or equivalent	None needed – no direct contact to sampling methods	No calibration required, only verification of proper function needed

B.5.2. Laboratory Instruments and Equipment

Laboratory instruments and equipment necessary for analytical measurement are presented in the laboratory SOPs (**Appendix B**).

Preventive maintenance of laboratory equipment generally will follow the guidelines recommended by the manufacturer. A malfunctioning instrument will be repaired immediately by in-house staff or through a service call from the manufacturer.

Maintenance schedules for laboratory equipment adhere to each manufacturer's recommendations. Records reflect the complete history of each instrument and specify the time frame for future maintenance. Major repairs or maintenance procedures are performed through service contracts with the manufacturer or qualified contractors. Paperwork associated with service calls and preventive maintenance calls will be kept on file by the laboratory.

Laboratory section managers are responsible for the routine maintenance of instruments used in the laboratory. Any routine preventive maintenance carried out is logged into the appropriate logbooks. The frequency of routine maintenance is dictated by the nature of samples being analyzed, the requirements of the method used and/or the

judgment of the laboratory section manager. Documentation procedures for instrument calibration and testing, including details of any observed problems, corrective measure(s), routine maintenance and instrument repair (including information regarding the repair and the individual who performed the repair).

All major instruments are backed up by comparable (if not equivalent) instrument systems in the event of unscheduled downtime. An inventory of spare parts is also available to minimize equipment/instrument downtime. In the event that the laboratory has an instrument problem which will prohibit the analysis of samples in a timely manner, an alternative laboratory may be considered.

On a daily basis, the operation of balances, incubators, ovens, refrigerators, and water purification systems will be checked and documented. Any discrepancies will be immediately reported to the appropriate laboratory personnel for resolution.

Instrument calibration will follow the specifications provided by the instrument manufacturer or specific analytical method used. When analyses are conducted according to USEPA methods, the calibration procedures and frequencies specified in the applicable method will be followed. Records of calibrations will be filed and maintained by the laboratory. These records will be subject to QA audit. For all instruments, the laboratory will maintain trained repair staff with in-house spare parts or will maintain service contracts with vendors.

All standards used to calibrate equipment are traceable, directly or indirectly, to the National Institute of Standards and Technology. All standards received will be logged into standard receipt logs maintained by the individual analytical groups. Each group will maintain a standards log that tracks the preparation of standards used for calibration and QC purposes.

B.6. Inspection/Acceptance of Supplies and Services

All supplies to be used in the field and laboratory will be available when needed. Field crews will inspect reusable and consumable field supplies before start of field activities daily and notify the field sampling lead to order additional supplies as necessary. Laboratory supplies will be inspected and sufficient stocks maintained by the subcontractor laboratory manager or delegate. Both field and laboratory supplies will be free of target chemicals and interferences, as verify through the use of equipment blanks. All reagents will be tested prior to use in the laboratory. All standards will be verified against a second source standard. The laboratory will follow a “first in/first out” procedure for the storage and use of all consumables to minimize the risk of contamination and degradation.

B.7. Environmental Information Management

The purpose of the data management system is to ensure that all of the necessary data are accurate and readily accessible to meet the analytical and reporting objectives of the project. The data management program established for the project includes field documentation and sample QA/QC procedures, methods for tracking and managing the data, and a system for filing all site-related information. More specifically, data management procedures will be employed to efficiently process the information collected such that the data are readily accessible and accurate. These procedures are described in detail in the following sections.

The data management plan also tracks the generation of the environmental data from its generation in the field through its final use and storage and consists of the following five elements: 1) sample designation system; 2) field activities; 3) sample tracking and management; 4) data management system; and 5) document control and inventory. These elements are described in more detail in the following sections.

B.7.1. Sample Designation System

A concise and easily understandable sample designation system is an important part of the project sampling activities. It provides a unique sample number that will facilitate both sample tracking and easy re-sampling of select locations to evaluate data gaps, if necessary. The sample designation system to be employed during the sampling activities will be consistent, yet flexible enough to accommodate unforeseen sampling events or conditions. A combination of letters and numbers will be used to yield a unique sample number for each field sampled collected, as outlined in **Section A.12.2**.

B.7.2. Field Activities

Field activities designed to gather the information necessary to make decisions require consistent documentation and accurate record keeping. During site activities, standardized procedures will be used for documentation of field activities, data security, and quality assurance. These procedures are described in further detail in the following subsections.

B.7.2.1. Field Documentation

Complete and accurate record keeping is a critical component of the field activities. When interpreting analytical results and identifying data trends, investigators realize that field notes are an important part of the review and validation process. To ensure that the field activities are thoroughly documented, several different information records, each with its own specific reporting requirements, will be maintained, including:

- Field logs;
- Chain of Custody forms; and
- Instrument Calibration Records.

A description of each of these types of field documentation is provided in **Section B.3**.

B.7.2.2. Data Security

Measures will be taken during the field activities to ensure that samples and records are not lost, damaged, or altered. When not in use, all field logbooks will be stored at the field office or locked in the field vehicle. Access to these files will be limited to the field personnel who utilize them. An electronic copy (e.g., scan to PDF) of all field data and laboratory data will be available to all project team members.

B.7.3. Sample Management and Tracking

A record of all field documentation will be maintained to ensure the validity of data used in the site analysis. To effectively execute such documentation, specific sample tracking and data management procedures will be used throughout the sampling activities.

As described in **Section B.3**, sample tracking will begin with the completion of chain of custody forms. The completed chain of custody forms associated with samples collected will be emailed to the project team members such as project manager, database manager, and QAM. Copies of all completed chain of custody forms will be maintained in the field office. The laboratory shall verify receipt of the samples electronically (via email) on the following day.

When analytical data are received from the laboratory, the project team will review the incoming analytical data packages against the information on the chain of custody forms to confirm that the correct analyses were performed for each sample and that results for all samples submitted for analysis were received. Any discrepancies noted will be promptly followed up by the project team.

B.7.4. Data Management System

In addition to the sample tracking system, a data management system will be implemented. The central focus of the data management system will be the development of a personal computer-based project database. The project database will combine pertinent geographical, field, and analytical data. Information that will be used to populate the database will be derived from three primary sources: surveying of sampling locations, field observations, and analytical results. Each of these sources is discussed in the following sections.

B.7.4.1. Computer Hardware

The database will be constructed on personal computer workstations connected through a Microsoft Exchange network server. The network will provide access to various hardware peripherals, such as laser printers, backup storage devices, image scanners, modems, etc. Computer hardware will be upgraded to industrial and corporate standards, as necessary, in the future.

B.7.4.2. Computer Software

The data will be maintained in the EQuIS data management system, currently at version 7.2 (updated with new versions over time). The database platform will be hosted in the cloud. Tables and other database reports will be generated through the EQuIS data management system in Microsoft Excel, ESRI Shape Files, or comma separated files as necessary depending on project requirements. These software products will be kept current in accordance with Arcadis practices regarding software versioning.

B.7.4.3. Survey Information

Investigation locations will be recorded with a GPS, and new monitoring wells will be surveyed by a state-certified land surveyor. Recording sample locations will ensure accurate documentation of sample locations for mapping purposes to facilitate the re-sampling of select sample locations during future investigative activities, if needed, and for any potential remediation activities. The surveying activities that will occur in the field will consist of the collection of information that will be used to compute a northing and easting in state plane coordinates (North American Datum of 1983 [NAD 83]) for each sample location and the collection of information to compute elevations relative to the National Geodetic Vertical Datum of 1929 for select sample locations, as appropriate. All field files associated with the surveying activities performed by Arcadis will be stored as a record of the project activities.

B.7.4.4. Field Observations

An important part of the information that will ultimately reside in the data management system for use during the project will originate in the observations that are recorded in the field.

Tables will be used to summarize measurements recorded in the field books or logs. It is anticipated that these tables will be developed using a personal computer spreadsheet program to reduce possible transcription error and to facilitate the transfer of information to the data management system. For example, for soil samples, the table would present the sampling date and time, sample location ID, sample collection depth, number of composited subsamples, and requested analyses.

All pertinent field data will be manually entered into the appropriate database tables from the chain of custody forms and field notebooks.

B.7.4.5. Analytical Results

Analytical results will be provided by the laboratory in a digital format. The data packages will be examined to ensure that the correct analyses were performed for each sample submitted and that all of the analyses requested on the chain of custody form were performed. If discrepancies are noted, the project manager, task leader, and QAM will be notified and will promptly follow up with the laboratory to resolve any issues.

Each data package will be validated. Any data that does not meet the specified standards will be flagged pending resolution of the issue. The flag will not be removed from the data until the issue associated with the sample results is resolved. Although flags may remain for certain data, the use of that data may not necessarily be restricted.

Following completion of the data validation, the digital files will be used to populate the appropriate database tables. The EDD format specifies one data record for each constituent for each sample analyzed. Specific fields include:

- Sample identification number;
- Date sampled;
- Date analyzed;
- Parameter name;
- Analytical result;
- Units;
- Detection limit; and
- Qualifier(s).

Any analytical data that cannot be provided by the laboratory in electronic format will be entered manually. After entry into the database, the EDD data will be compared to the field information previously entered into the database to confirm that all requested analytical data have been received.

B.7.4.6. Data Analysis and Reporting

The database management system will have several functions to facilitate the review and analysis of the data. Data entry screens will be developed to assist in the keypunching of field observations. Routines will also be developed to permit the user to scan analytical data from a given site for a given media. Several output functions that have been developed by Arcadis will be appropriately modified for use in the data management system.

A valuable function of the data management system will be the generation of tables of analytical results from the project databases. The capability of the data management system to directly produce tables reduces the redundant manual entry of analytical results during report preparation and precludes transcription errors that may occur otherwise. This data management system has the ability to create tables of analytical data to use as part of data interpretation tasks, reporting of data, and generation of future reporting.

Another function of the data management system will be to create digital files of analytical results and qualifiers suitable for transfer to mapping/presentation software. A function has been created by Arcadis that creates a digital file consisting of sample location number, state plane coordinates, sampling date, and specified

constituents with associated concentrations and analytical qualifiers. The file is then transferred to an AutoCAD and or geographical information system workstation, where another program has been developed to plot a location's analytical data in a "box" format at the sample location (represented by the state plane coordinates). This routine greatly reduces the redundant keypunching of analytical results and facilitates the efficient production of interpretative and presentation graphics.

The data management system also has the capability of producing a digital file of select parameters that exists in one or more of the databases. This type of custom function is accomplished on an interactive basis and is best used for transferring select information into a number of analysis tools, such as statistical or graphing programs.

B.7.5. Document Control and Inventory

Arcadis maintains project files at its Melville, New York office as discussed in **Section A.12.6**.

C. Group C: Assessment, Response Actions, and Oversight

This section describes how activities will be checked to ensure that they are completed correctly and according to procedures outlined in this QAPP. Types of planned assessments, response actions, and project oversight activities are discussed below.

C.1. Assessment and Response Actions

Types of assessments planned relevant to this QAPP include field and laboratory audits which are described below. Response actions to be taken in response to the audits are also described in this section.

C.1.1. Field Audits

All field personnel will be responsible for correct implementation of the sampling protocols. Daily meetings will be held with all field personnel before the start of field activities to ensure objectives and protocols are understood. When conducting the field work, if a condition is noted by the field crew that would have an adverse effect on data quality, corrective action will be taken as soon as feasible so as not to repeat this condition (see **Section C.1.3**). Any deviations to the field SOPs or procedures outlined in the QAPP will be documented in the field logbook and discussed with the field sampling lead.

In addition, systems audits comparing scheduled QA/QC activities from this QAPP with actual QA/QC activities completed will be performed. The project manager and QAM may periodically confirm that work is being performed consistent with this QAPP.

C.1.2. Laboratory Audits

Internal laboratory audits are conducted by the laboratory QA/QC officer consistent with their Quality Systems Manual. As part of the audit, the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the laboratory quality assurance manual and SOPs. The results of the audits are summarized and issued to each department supervisor, the laboratory manager, and the laboratory director. A systems audit of each laboratory is also performed by the QAM to determine if the procedures implemented by each laboratory are in compliance with the QAPP and SOPs.

In addition to the laboratory's internal audits, as participants in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification. Audits are usually conducted on an annual basis and focus on laboratory conformance to the specific program protocols for which the laboratory is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation, and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action, if necessary.

Arcadis reserves the right to conduct an on-site audit of the laboratory during the course of the project, as deemed necessary.

C.1.3. Corrective Actions

Corrective actions are required when field or analytical data are not within the objectives specified in this QAPP. Corrective actions include procedures to promptly investigate, document, evaluate, and correct data collection and/or analytical procedures. Field and laboratory corrective action procedures for the actions are described below.

C.1.3.1. Field Procedures

When conducting the field work, if a condition is noted by the field crew that would have an adverse effect on data quality, corrective action will be initiated upon identification of the problem so as not to repeat this condition. Condition identification, cause, and corrective action implemented by the task leader or a designee, will be documented on a Corrective Action Form and reported to the appropriate Arcadis task leader, QAM, and project manager.

Examples of situations that would require corrective actions are provided below:

- Protocols as defined by the QAPP have not been followed;
- Equipment is not in proper working order;
- QC requirements have not been met; or
- Issues resulting from performance or systems audits have not been resolved.

Project personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities.

C.1.3.2. Laboratory Procedures

In the laboratory, when a condition is noted to have an adverse effect on data quality, corrective action will be initiated upon identification of the problem so as not to repeat this condition. Condition identification, cause, and corrective action taken will be documented and reported to the project manager and QAM.

Corrective action may be initiated, at a minimum, under the following conditions:

- Specific laboratory analytical protocols have not been followed;
- Protocols as defined by this QAPP have not been followed;
- Predetermined data acceptance standards are not obtained;
- Equipment is not in proper working order or calibrated;
- Sample and test results are not completely traceable;
- QC requirements have not been met; or

- Issues resulting from performance or systems audits have not been resolved.

Laboratory personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities. Corrective action is initiated at a point where the problem has been identified. At whatever level this occurs (analyst, supervisor, data review, or quality control), it is brought to the attention of the laboratory QA/QC manager and, ultimately, the laboratory director. Final approval of any action deemed necessary is subject to the approval of the laboratory director.

Any corrective action deemed necessary based on system or performance audits or the results of data review will be documented and implemented. The corrective action may include sample re-extraction, re-preparation, re-analysis, cleanup, dilutions, matrix modifications, or other activities.

C.2. Oversight and Reports to Management

Oversight activities ensure that response actions and reporting mechanisms are in place to capture the project status and any QA issues that arise during implementation and through assessments. Project oversight and reporting are discussed below.

C.2.1. Project Oversight

The Contractor project manager will be responsible for overall project oversight as well as. The QAM will ensure that reports generated for the project will also include documentation for any corrective actions implemented in the field and/or laboratory.

C.2.2. Internal Reporting

The analytical laboratory will submit analytical reports to Arcadis for review. If required, Arcadis will, in turn, submit the reports to the data validator for review. Supporting data (i.e., historical data, related field or laboratory data) will also be reviewed to evaluate data quality, as appropriate. The Arcadis QAM will incorporate results of the data validation reports (if required) and assessments of data usability into a summary report (if required) that will be submitted to the Arcadis project manager and appropriate task leader. If required, this report will be filed in the project file at the Arcadis office and will include the following:

- Assessment of data accuracy, precision, and completeness for both field and laboratory data;
- Results of the performance and systems audits;
- Significant QA/QC problems, solutions, corrections, and potential consequences; and
- Analytical data validation report.

C.2.3. Reporting

Upon sample transport to the laboratory, a copy of the chain of custody will be forwarded to the Arcadis project manager. Upon receipt of the data package from the laboratory, the Arcadis QAM will determine if the data package has met the required data quality objectives. The analytical data package will be submitted to the Arcadis project manager and the analytical data will be incorporated into a tabulated format.

D. Group D: Environmental Information Review and Usability Determination

This section addresses the activities associated with environmental information review for the purpose of determining whether the environmental information meets the established environmental/data quality objectives, the performance/acceptance criteria, and are useable for its intended purpose. Information review activities ensure that products and services resulting from the environmental information operations are of known and documented quality for their intended use(s) and that any limitations concerning its intended use is documented and communicated.

D.1. Environmental Information Review

After field and laboratory data are obtained, the data subject to a review and data reduction process to ensure correct and accurate data reporting. The data will be subject to the following:

- Reduction, or manipulation mathematically, or otherwise into meaningful and useful forms;
- Review; and
- Organization, interpretation, and reporting

Following data reduction and review, the data will also be subject to the processes in this section to ensure that the data are verified and validated and the DQOs are met and data quality is sufficient for the intended uses of the data. As described by USEPA (2023):

- Data Verification is the process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual requirements.
- Data Validation is an analyte- and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance (i.e., data verification) to determine the analytical quality of a specific data set.
- Data Quality Assessment is the scientific and statistical evaluation of data to determine if the data obtained from environmental information operations are of the right type, quality, and quantity to support their intended use.

D.1.1. Field Data Reduction and Review

D.1.1.1. Field Data Reduction

Information collected in the field through visual observation and/or manual measurement will be recorded in field logbooks, data sheets, and/or forms. Such data will be reviewed by the appropriate task leader for adherence to this QAPP and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible, and, as necessary, incorporated into the data evaluation process.

D.1.1.2. Field Data Review

Field data review will be conducted by the field personnel and reviewed for accuracy by the appropriate task leader and the QAM. Logs and documents will be checked for:

- General completeness;

- Readability;
- Usage of appropriate procedures;
- Reasonableness in comparison to present and past data collected; and
- Correct sample locations.

D.1.2. Laboratory Data Reduction and Review

D.1.2.1. Laboratory Data Reduction

The calculations used for data reduction will be specified in each of the analytical methods referenced previously. Whenever possible, analytical data will be transferred directly from the instrument to a computerized data system. Raw data will be entered into permanently bound laboratory notebooks. The data entered are sufficient to document all factors used to arrive at the reported value.

The calculations used for laboratory data reduction will be in accordance with the analytical methods. Concentration calculations for chromatographic analyses will be based on the appropriate method. Quantitation will be performed using either internal or external standards.

Results will be reported on a dry-weight basis. Unless otherwise specified, all values will be reported uncorrected for blank contamination.

D.1.2.2. Laboratory Data Review

Data will be subject to multi-level review by the laboratory. The group leader will review all data reports prior to release for final data report generation. The laboratory QA/QC manager will review the final data reports and will review a cross-section of the final data reports prior to shipment to Arcadis. Primary analysts review the data that they produce, and a secondary data review is performed by secondary analysts and/or their supervisors.

If discrepancies or deficiencies exist in the analytical results, then corrective action will be taken, as discussed in **Section C.1.3.2**. Deficiencies discovered as a result of internal data review, as well as the corrective actions to be used to rectify the situation, will be documented on a Corrective Action Form. This form will be submitted to the Arcadis project manager and QAM.

D.1.3. Data Verification and Validation

All data generated for use in making decisions regarding protection of human health or the environment and for engineering design/control purposes will be subjected to the data validation and verification procedures outlined in this section. Data generated for qualitative purposes, disposal purposes and geotechnical testing will not be reviewed.

As described by USEPA (2002b), data verification is a completeness check that all specified activities involved in data collection and processing have been completed and documented and that the necessary records (objective evidence) are available to proceed to data validation. Data verification entails a review of the quality control data and the raw data to verify that the laboratory was operating within required limits, the analytical results were correctly transcribed from the instrument read outs, and which, if any, environmental samples were related to any out-of-control quality control samples. The objective of data validation is to identify any questionable or invalid laboratory measurements. Data verification will occur throughout the environmental information operations, including in the field or within the laboratory at each level (i.e., all personnel should verify their own work) and data verification should also occur as information is passed from one level to the next (i.e., the sample custodian

should verify the information provided by the field personnel, and supervisors should verify the information produced by their staff). Additionally, data verification will be completed by the QAC after receipt of field records or a complete data package. To the extent possible, records are reviewed for completeness, for factual content, and against project specifications (e.g., SOPs, Work Plan, FSP, QAPP). The QAM will use the performance standards described in **Section A.6.2** and assessment methods described in **Section B.4.4** will be used to verify the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual requirements.

As described by USEPA (2002b), data validation is a standardized review process for judging the analytical quality and usefulness of a discrete set of chemical data and is necessary to ensure that data of known and documented quality are used in making environmental decisions that meet the DQOs of the site. Data validation is a systematic process that compares a body of data to the requirements in a set of documented acceptance criteria to ascertain its completeness, correctness, and consistency. The data validation personnel will work independently from all other project teams. Data validators will not be involved in sampling or data analysis/reporting for the end user.

Arcadis will validate all data generated producing a data review report for each individual SDG using the USEPA's National Function Guidelines outlined below. These procedures and criteria may be modified as necessary to address project-specific and method-specific criteria, control limits, and procedures. Data validation will consist of data screening, checking, reviewing, editing, and interpretation to document analytical data quality and to determine whether the quality is sufficient to meet the DQOs using analytical data verification and validation stage approach, adopted from the "Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use" (January 2009), level of data validation as outlined below (USEPA 2009).

The data validator will verify that reduction of laboratory measurements and laboratory reporting of analytical parameters is in accordance with the procedures specified for each analytical method and/or as specified in this QAPP. Any deviations from the analytical method or any special reporting requirements apart from that specified in this QAPP will be detailed on chain of custody forms.

Data validation procedures shall be performed for both laboratory operations. Validation of the analytical data will be performed by the QAM or designee using the following documents as guidance for the review process:

- USEPA National Functional Guidelines for Organic Superfund Methods Data Review, EPA 540-R-20-005, November 2020 (with reference to the historical USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, OSWER 9240.1-05A-P, October 1999, as appropriate).
- USEPA National Functional Guidelines for Inorganic Superfund Methods Data Review, EPA 542-R-20-006, November 2020 (with reference to the historical USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, OSWER 9240.1-45, October 2004, as appropriate).
- United States Department of Defense Data Validation Guidelines Module 6: Data Validation Procedure for PFAS by Quality Systems Manual Table B-24, October 2022.

Validation will be performed on the complete data set following the Data Usability Summary Report (DUSR) criteria set forth in NYSDEC DER-10 as a Stage 3 data validation. Data verification and validation includes review of the following elements:

- Documentation identifies laboratory receiving and conducting analyses and includes documentation for all samples submitted by the project.
- Requested analytical methods were performed and analysis dates are present.

- Requested target analyte results are reported along with laboratory data qualifiers and data qualifier definitions.
- Requested target analyte result units are reported.
- Requested reporting limits for all samples are reported and results at and below the requested reporting limits are clearly identified.
- Sampling dates, date and time of laboratory receipt of samples, and sample conditions upon receipt at the laboratory.
- Sample-related QC results and acceptance criteria are provided and linked to the reported field samples, and frequency is appropriate.
- Technical holding times are evaluated.
- Initial calibration, initial calibration verification, and continuing calibration verification data are provided and analyzed at the required frequency.
- Method specific instrument performance checks are present, appropriate, and analyzed at the required frequency.
- Sample results are evaluated and qualified by comparing holding times and sample-related and instrument-related QC data to the requirements and guidelines presented in this QAPP and the data validation guidelines presented in the documents referenced above.
- Instrument response data are reported and associated with appropriate internal standards and meet the requirements for each analyte.
- Raw data reported and spot checked.
- Confirmation of calculation of each parameter group within ten percent of total number of laboratory SDGs.

The completeness of each data package will be evaluated by the data validator. Completeness checks will be administered on all data to determine whether deliverables specified in the QAPP are present. At a minimum, deliverables will include sample chain of custody forms, analytical results, QC summaries and supporting raw data from instrument printouts. The review will determine whether all required items are present and request copies of missing deliverables.

Deficiencies discovered as a result of the data review, as well as the corrective actions implemented in response, will be documented and submitted in the form of a written report addressing the following topics as applicable to each method:

- Assessment of the data package;
- Description of any protocol deviations;
- Failures to reconcile reported and/or raw data;
- Assessment of any compromised data;
- Overall appraisal of the analytical data; and
- Table of site name, sample quantities, matrix, and fractions analyzed.

During the review process, qualified and unqualified data are verified against the supporting documentation. Based on this evaluation, qualifier codes may be added, deleted, or modified by the data reviewer. Results will be qualified with the following codes in accordance with the USEPA National Functional Guidelines:

Qualifier	Definition
Concentration (C) Qualifiers (Laboratory Assigned)	
U	The analyte/compound was analyzed for but not detected. The associated value is the compound reporting limit.
B	The analyte/compound has been found in the sample as well as its associated blank, its presence in the sample may be suspect. (Note: laboratory assigned B qualifiers will be removed during data validation if the concentration detected in the sample is greater than five-times the concentration detected in the associated blank.)
J	The analyte/compound was positively identified; however, the associated numerical value is an estimated concentration only.
Quantitation (Q) Qualifiers (Laboratory Assigned)	
E	The compound was quantitated above the calibration range.
D	Concentration is based on a diluted sample analysis.
Validation Qualifiers	
UJ	The analyte/compound was not detected above the reported sample quantitation limit; however, the reported limit is approximate and may or may not represent the actual reporting limit.
UB	The analyte/compound is considered non-detect at the listed value due to associated blank contamination (i.e., concentration in the sample is less than five-times the concentration detected in the associated blank).
J	The analyte/compound was positively identified; however, the associated numerical value is an estimated concentration.
R	The sample results are rejected.

It should be noted that qualified results do not necessarily invalidate data. The goal to produce the best possible data does not necessarily mean producing data without quality control qualifiers. Qualified data can provide useful information.

Resolution of any issues regarding laboratory performance or deliverables will be handled between the laboratory and the data validator. Suggestions for reanalysis may be made by the Arcadis QAM at this point.

Data validation reports are signed by the validator and peer reviewer. Data validation reports will be kept in the electronic project file at the Arcadis office in Melville, New York, as well as included in the final report deliverable to the agency.

D.2. Usability Determination

This section described the data useability determination, is performed at the conclusion of the environmental information operations using the outputs of the environmental information/data verification, the environmental information/data validation, and the environmental information/data quality assessment activities. This reconciliation phase involves a qualitative and quantitative evaluation of environmental information to determine if

the project information is of the right type, quality, and quantity to support its intended use and are suitable for the decisions that will be made.

D.2.1. Reconciliation with User Requirements

The data results will be examined to determine the performance achieved for each DQI. The performance will then be compared with the project objective and DQOs. Of note will be sample results at or near action levels. All deviations from objectives will be noted. Additional action may be warranted when performance does not meet performance objectives for critical data. Options for corrective action relating to incomplete information, questionable results, or inconsistent data may include the following:

- Retrieval of missing information;
- Request for additional explanation or clarification;
- Reanalysis of sample from extract (when appropriate); and
- Recalculation or reinterpretation of results by the laboratory.

These actions may improve the data quality, reduce uncertainty, and may eliminate the need to qualify or reject data. If these actions do not improve the data quality to an acceptable level, the following additional actions may be taken:

- Extrapolation of missing data from existing data points;
- Use of historical data; and
- Evaluation of the critical/non-critical nature of the sample.

If the data gap cannot be resolved by these actions, an evaluation of the data bias and potential for false negatives and positives can be evaluated. If the resultant uncertainty level is unacceptable, additional sample collection and analysis.

D.2.2. Reconciliation with Data Quality Objectives

The investigation goals include data completeness and acceptance criteria as listed above in **Section A.6.2**. If these criteria are not met, data useability for purposes of meeting the DQOs will be determined by evaluating the ability of the final dataset to meet the investigation goals.

E. References

USEPA. 1992a. Guide to Management of Investigation-Derived Wastes. OSWER Directive 9345.3-03FS. January 15, 1992.

USEPA 1992b. Specifications and Guidance for Contaminant-Free Sample Containers. OSWER Directive 9240.05A, EPA/540/R-93/051. December 1992.

USEPA. 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process. EPA/240/B-06/001. February.

USEPA. 2023. Quality Assurance Project Plan Standard. CIO 2105.S-02.0. July.

Tables

Table 1a
Parameters, Methods, and Target Reporting Limits
Quality Assurance Project Plan
Long Island MacArthur Airport
Ronkonkoma, New York

Analyte	CAS number	Units	Restricted Use Soil Cleanup Objectives[1]			Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
			Commercial	Industrial	Protection of Groundwater	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Per- and Polyfluoroalkyl Substances (Method EPA 1633)											
Perfluorobutanoic acid (PFBA)	375-22-4	ng/g	NS	NS	NS	0.80	0.10	0.80	0.11	--	--
Perfluoropentanoic acid (PFPeA)	2706-90-3	ng/g	NS	NS	NS	0.40	0.10	0.40	0.070	--	--
Perfluorohexanoic acid (PFHxA)	307-24-4	ng/g	NS	NS	NS	0.20	0.090	0.20	0.030	--	--
Perfluoroheptanoic acid (PFHpA)	375-85-9	ng/g	NS	NS	NS	0.20	0.090	0.20	0.050	--	--
Perfluorooctanoic acid (PFOA)	335-67-1	ng/g	500	600	0.8	0.20	0.051	0.20	0.080	--	--
Perfluorononanoic acid (PFNA)	375-95-1	ng/g	NS	NS	NS	0.20	0.050	0.20	0.050	--	--
Perfluorodecanoic acid (PFDA)	335-76-2	ng/g	NS	NS	NS	0.20	0.050	0.20	0.070	--	--
Perfluoroundecanoic acid (PFUnA)	2058-94-8	ng/g	NS	NS	NS	0.20	0.050	0.20	0.060	--	--
Perfluorododecanoic acid (PFDoA)	307-55-1	ng/g	NS	NS	NS	0.20	0.050	0.20	0.080	--	--
Perfluorotridecanoic acid (PFTrDA)	72629-94-8	ng/g	NS	NS	NS	0.20	0.050	0.20	0.090	--	--
Perfluorotetradecanoic acid (PFTeDA)	376-06-7	ng/g	NS	NS	NS	0.20	0.050	0.20	0.10	--	--
Perfluorobutanesulfonic acid (PFBS)	375-73-5	ng/g	NS	NS	NS	0.20	0.050	0.20	0.070	--	--
Perfluoropentanesulfonic acid (PFPeS)	2706-91-4	ng/g	NS	NS	NS	0.20	0.050	0.20	0.10	--	--
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	ng/g	NS	NS	NS	0.20	0.050	0.20	0.11	--	--
Perfluoroheptanesulfonic acid (PFHpS)	375-92-8	ng/g	NS	NS	NS	0.20	0.050	0.20	0.10	--	--
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	ng/g	440	440	1	0.50	0.21	0.20	0.050	--	--
Perfluorononanesulfonic acid (PFNS)	68259-12-1	ng/g	NS	NS	NS	0.20	0.050	0.20	0.14	--	--
Perfluorododecanesulfonic acid (PFDoS)	79780-39-5	ng/g	NS	NS	NS	0.20	0.050	0.20	0.050	--	--
1H,1H,2H,2H-Perfluorohexane sulfonic acid (4:2 FTS)	757124-72-4	ng/g	NS	NS	NS	0.80	0.20	0.80	0.28	--	--
1H,1H,2H,2H-Perfluorooctane sulfonic acid (6:2 FTS)	27619-97-2	ng/g	NS	NS	NS	1.0	0.35	0.80	0.10	--	--
1H,1H,2H,2H-Perfluorodecane sulfonic acid (8:2 FTS)	39108-34-4	ng/g	NS	NS	NS	1.0	0.35	0.80	0.19	--	--
Perfluorooctanesulfonamide (PFOSA)	754-91-6	ng/g	NS	NS	NS	0.20	0.090	0.20	0.070	--	--
N-methylperfluorooctane sulfonamide (NMeFOSA)	31506-32-8	ng/g	NS	NS	NS	0.20	0.050	0.20	0.060	--	--
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	2355-31-9	ng/g	NS	NS	NS	0.20	0.050	0.20	0.080	--	--
N-ethylperfluorooctane sulfonamide (NEtFOSA)	4151-50-2	ng/g	NS	NS	NS	0.20	0.050	0.20	0.050	--	--
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	2991-50-6	ng/g	NS	NS	NS	0.20	0.050	0.20	0.080	--	--
N-methylperfluorooctane sulfonamidoethanol (NMeFOSE)	24448-09-7	ng/g	NS	NS	NS	2.0	0.50	0.20	0.13	--	--
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	1691-99-2	ng/g	NS	NS	NS	2.0	0.50	2.00	0.28	--	--
Hexafluoropropylene Oxide Dimer Acid (HFPO-DA)	13252-13-6	ng/g	NS	NS	NS	0.80	0.30	2.00	0.73	--	--
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	919005-14-4	ng/g	NS	NS	NS	0.80	0.20	0.80	0.18	--	--
Perfluoro-4-methoxybutanoic acid (PFMBA)	863090-89-5	ng/g	NS	NS	NS	0.40	0.10	0.80	0.12	--	--
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	151772-58-6	ng/g	NS	NS	NS	0.40	0.10	0.40	0.13	--	--
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	756426-58-1	ng/g	NS	NS	NS	0.80	0.20	0.80	0.26	--	--
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	763051-92-9	ng/g	NS	NS	NS	0.80	0.20	0.80	0.18	--	--
Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)	113507-82-7	ng/g	NS	NS	NS	0.40	0.10	0.40	0.030	--	--
3-Perfluoropropylpropanoic acid (3:3 FTCA)	356-02-5	ng/g	NS	NS	NS	1.0	0.25	1.00	0.24	--	--
3-Perfluoropentylpropanoic acid (5:3 FTCA)	914637-49-3	ng/g	NS	NS	NS	5.0	1.0	5.00	0.62	--	--
3-Perfluoroheptylpropanoic acid (7:3 FTCA)	812-70-4	ng/g	NS	NS	NS	5.0	1.0	5.00	0.63	--	--
Perfluorodecanesulfonic acid (PFDS)	335-77-3	ng/g	NS	NS	NS	0.20	0.050	0.20	0.11	--	--
Perfluoro-3-methoxypropanoic acid (PFMPA)	377-73-1	ng/g	NS	NS	NS	0.40	0.10	0.40	0.030	--	--

Table 1a
Parameters, Methods, and Target Reporting Limits
Quality Assurance Project Plan
Long Island MacArthur Airport
Ronkonkoma, New York

Analyte	CAS number	Units	Restricted Use Soil Cleanup Objectives[1]			Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
			Commercial	Industrial	Protection of Groundwater	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Per- and Polyfluoroalkyl Substances (Method ASTM D8421)											
Perfluorobutanoic acid (PFBA)	375-22-4	µg/Kg	NS	NS	NS	--	--	400	70	0.5	0.01348
Perfluoropentanoic acid (PFPeA)	2706-90-3	µg/Kg	NS	NS	NS	--	--	200	70	0.5	0.03089
Perfluorohexanoic acid (PFHxA)	307-24-4	µg/Kg	NS	NS	NS	--	--	100	30	0.1	0.00354
Perfluoroheptanoic acid (PFHpA)	375-85-9	µg/Kg	NS	NS	NS	--	--	100	30	0.1	0.00972
Perfluorooctanoic acid (PFOA)	335-67-1	µg/Kg	500	600	0.8	--	--	100	40	0.1	0.01324
Perfluorononanoic acid (PFNA)	375-95-1	µg/Kg	NS	NS	NS	--	--	100	25	0.1	0.00599
Perfluorodecanoic acid (PFDA)	335-76-2	µg/Kg	NS	NS	NS	--	--	100	50	0.1	0.0145
Perfluoroundecanoic acid (PFUnA)	2058-94-8	µg/Kg	NS	NS	NS	--	--	100	25	0.1	0.00785
Perfluorododecanoic acid (PFDoA)	307-55-1	µg/Kg	NS	NS	NS	--	--	100	50	0.1	0.00611
Perfluorotridecanoic acid (PFTrDA)	72629-94-8	µg/Kg	NS	NS	NS	--	--	100	75	0.1	0.00826
Perfluorotetradecanoic acid (PFTeDA)	376-06-7	µg/Kg	NS	NS	NS	--	--	100	55	0.5	0.5
Perfluorobutanesulfonic acid (PFBS)	375-73-5	µg/Kg	NS	NS	NS	--	--	100	70	0.1	0.00727
Perfluoropentanesulfonic acid (PFPeS)	2706-91-4	µg/Kg	NS	NS	NS	--	--	100	55	0.1	0.00663
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	µg/Kg	NS	NS	NS	--	--	100	60	0.1	0.00445
Perfluoroheptanesulfonic acid (PFHpS)	375-92-8	µg/Kg	NS	NS	NS	--	--	100	55	0.1	0.00631
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	µg/Kg	440	440	1	--	--	100	40	0.1	0.00397
Perfluorononanesulfonic acid (PFNS)	68259-12-1	µg/Kg	NS	NS	NS	--	--	100	70	0.1	0.00465
Perfluorododecanesulfonic acid (PFDoS)	79780-39-5	µg/Kg	NS	NS	NS	--	--	100	95	0.5	0.5
1H,1H,2H,2H-Perfluorohexane sulfonic acid (4:2 FTS)	757124-72-4	µg/Kg	NS	NS	NS	--	--	400	340	0.1	0.01008
1H,1H,2H,2H-Perfluorooctane sulfonic acid (6:2 FTS)	27619-97-2	µg/Kg	NS	NS	NS	--	--	400	200	0.2	0.03453
1H,1H,2H,2H-Perfluorodecane sulfonic acid (8:2 FTS)	39108-34-4	µg/Kg	NS	NS	NS	--	--	400	240	0.1	0.02314
Perfluorooctanesulfonamide (PFOSA)	754-91-6	µg/Kg	NS	NS	NS	--	--	100	35	0.1	0.00308
N-methylperfluorooctane sulfonamide (NMeFOSA)	31506-32-8	µg/Kg	NS	NS	NS	--	--	100	90	0.1	0.00576
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	2355-31-9	µg/Kg	NS	NS	NS	--	--	100	40	0.1	0.01052
N-ethylperfluorooctane sulfonamide (NEtFOSA)	4151-50-2	µg/Kg	NS	NS	NS	--	--	100	90	0.1	0.00421
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	2991-50-6	µg/Kg	NS	NS	NS	--	--	130	110	0.1	0.01288
N-methylperfluorooctane sulfonamidoethanol (NMeFOSE)	24448-09-7	µg/Kg	NS	NS	NS	--	--	1000	280	0.1	0.01259
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	1691-99-2	µg/Kg	NS	NS	NS	--	--	1000	300	0.1	0.00377
Hexafluoropropylene Oxide Dimer Acid (HFPO-DA)	13252-13-6	µg/Kg	NS	NS	NS	--	--	400	190	0.1	0.00397
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	919005-14-4	µg/Kg	NS	NS	NS	--	--	400	200	0.1	0.0052
Perfluoro-4-methoxybutanoic acid (PFMBA)	863090-89-5	µg/Kg	NS	NS	NS	--	--	800	320	0.1	0.00314
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	151772-58-6	µg/Kg	NS	NS	NS	--	--	800	420	0.1	0.00794
9-Chlorohexadecafluoro-3-oxananone-1-sulfonic acid (9Cl-PF3ONS)	756426-58-1	µg/Kg	NS	NS	NS	--	--	400	220	0.1	0.0036
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	763051-92-9	µg/Kg	NS	NS	NS	--	--	400	160	0.1	0.00433
Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)	113507-82-7	µg/Kg	NS	NS	NS	--	--	400	190	0.1	0.00458
3-Perfluoropropylpropanoic acid (3:3 FTCA)	356-02-5	µg/Kg	NS	NS	NS	--	--	500	140	0.4	0.01356
3-Perfluoropentylpropanoic acid (5:3 FTCA)	914637-49-3	µg/Kg	NS	NS	NS	--	--	2500	870	0.4	0.01782
3-Perfluoroheptylpropanoic acid (7:3 FTCA)	812-70-4	µg/Kg	NS	NS	NS	--	--	2500	810	0.4	0.01265
Perfluorodecanesulfonic acid (PFDS)	335-77-3	µg/Kg	NS	NS	NS	--	--	100	45	0.1	0.00618
Perfluoro-3-methoxypropanoic acid (PFMPA)	377-73-1	µg/Kg	NS	NS	NS	--	--	800	410	0.1	0.0034

Table 1a
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Quality Assurance Project Plan
Long Island MacArthur Airport
Ronkonkoma, New York

ARCADIS Design & Consultancy for natural and built assets

Analyte	CAS number	Units	Restricted Use Soil Cleanup Objectives[2]			Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
			Commercial	Industrial	Protection of Groundwater	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Volatile Organic Compounds (Method SW-846 8260D)											
1,1,1-Trichloroethane	71-55-6	µg/Kg	500,000	1,000,000	680	50	14	2	0.48	--	--
1,1,2,2-Tetrachloroethane	79-34-5	µg/Kg	NS	NS	600	50	9.91	2	0.6	--	--
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	µg/Kg	NS	NS	6,000	50	17	5	2.7	--	--
1,1,2-Trichloroethane	79-00-5	µg/Kg	NS	NS	NS	50	10.2	2	0.55	--	--
1,1-Dichloroethane	75-34-3	µg/Kg	240,000	480,000	270	50	12	1	0.5	--	--
1,1-Dichloroethene	75-35-4	µg/Kg	500,000	1,000,000	330	50	13.2	1	0.66	--	--
1,2,3-Trichlorobenzene	87-61-6	µg/Kg	NS	NS	NS	50	17.5	5	2.5	--	--
1,2,4-Trichlorobenzene	120-82-1	µg/Kg	NS	NS	3,400	50	13.5	5	2.5	--	--
1,2-Dibromo-3-chloropropane	96-12-8	µg/Kg	NS	NS	NS	50	10.6	2	0.69	--	--
1,2-Dichlorobenzene	95-50-1	µg/Kg	500,000	1,000,000	1,100	50	11	1	0.55	--	--
1,2-Dichloroethane	107-06-2	µg/Kg	30,000	60,000	20	50	12.5	1	0.47	--	--
1,2-Dichloropropane	78-87-5	µg/Kg	NS	NS	NS	50	9.0	2	0.47	--	--
1,3-Dichlorobenzene	541-73-1	µg/Kg	280,000	560,000	2,400	50	16.5	1	0.5	--	--
1,4-Dichlorobenzene	106-46-7	µg/Kg	130,000	250,000	1,800	50	16.7	1	0.49	--	--
Methyl ethyl ketone-MEK (2-Butanone)	78-93-3	µg/Kg	500,000	1,000,000	300	250	110	10	2.4	--	--
2-Hexanone	591-78-6	µg/Kg	NS	NS	NS	250	56.8	5	2.1	--	--
4-Methyl-2-pentanone (MIBK)	108-10-1	µg/Kg	NS	NS	1,000	250	65.1	5	2.3	--	--
Acetone	67-64-1	µg/Kg	500,000	1,000,000	50	250	221	10	4.1	--	--
Benzene	71-43-2	µg/Kg	44,000	89,000	60	50	10.1	0.5	0.46	--	--
Bromoform	75-25-2	µg/Kg	NS	NS	NS	50	9	5	1.4	--	--
Bromomethane	74-83-9	µg/Kg	NS	NS	NS	50	27.7	5	0.76	--	--
Carbon disulfide	75-15-0	µg/Kg	NS	NS	2,700	50	33.7	2	0.54	--	--
Carbon tetrachloride	56-23-5	µg/Kg	22,000	44,000	760	50	16.5	2	0.62	--	--
Chlorobenzene	108-90-7	µg/Kg	500,000	1,000,000	1,100	50	12	2	0.46	--	--
Chlorobromomethane	74-97-5	µg/Kg	NS	NS	NS	50	15	5	0.56	--	--
Chlorodibromomethane	124-48-1	µg/Kg	NS	NS	NS	50	11	2	0.56	--	--
Chloroethane	75-00-3	µg/Kg	NS	NS	1,900	50	18.5	5	0.59	--	--
Chloroform	67-66-3	µg/Kg	350,000	700,000	370	50	11	2	1	--	--
Chloromethane	74-87-3	µg/Kg	NS	NS	NS	50	20.1	5	2	--	--
cis-1,2-Dichloroethene	156-59-2	µg/Kg	500,000	1,000,000	250	50	13	1	0.84	--	--
cis-1,3-Dichloropropene	10061-01-5	µg/Kg	NS	NS	NS	50	11.1	2	0.48	--	--
Cyclohexane	110-82-7	µg/Kg	NS	NS	NS	50	13	2	0.66	--	--
Dichlorobromomethane	75-27-4	µg/Kg	NS	NS	NS	50	7.5	2	0.43	--	--
Dichlorodifluoromethane	75-71-8	µg/Kg	NS	NS	NS	50	15.5	5	0.73	--	--
Ethylbenzene	100-41-4	µg/Kg	390,000	780,000	1,000	50	15	1	0.45	--	--
Ethylene Dibromide	106-93-4	µg/Kg	NS	NS	NS	50	9.5	1	0.42	--	--
Isopropylbenzene	98-82-8	µg/Kg	NS	NS	2,300	50	16	2	1.4	--	--
Methyl acetate	79-20-9	µg/Kg	NS	NS	NS	250	39.2	5	1.4	--	--
Methyl tert-Butyl Ether (MTBE)	1634-04-4	µg/Kg	500,000	1,000,000	930	50	10.7	1	0.47	--	--
Methylcyclohexane	108-87-2	µg/Kg	NS	NS	NS	50	36.3	2	0.88	--	--
Methylene Chloride	75-09-2	µg/Kg	500,000	1,000,000	50	50	10.5	5	2.6	--	--
m-Xylene & p-Xylene	179601-23-1	µg/Kg	500,000	1,000,000	1,600	50	14	1	0.9	--	--
o-Xylene	95-47-6	µg/Kg				50	16	1	0.46	--	--
Styrene	100-42-5	µg/Kg	NS	NS	NS	50	8.5	2	0.4	--	--
Tetrachloroethene	127-18-4	µg/Kg	150,000	300,000	1,300	50	18	2	0.58	--	--
Toluene	108-88-3	µg/Kg	500,000	1,000,000	700	50	12.5	1	0.53	--	--
trans-1,2-Dichloroethene	156-60-5	µg/Kg	500,000	1,000,000	190	50	9	1	0.61	--	--
trans-1,3-Dichloropropene	10061-02-6	µg/Kg	NS	NS	NS	50	11.1	2	0.46	--	--
Trichloroethene	79-01-6	µg/Kg	200,000	400,000	470	50	11	1	0.76	--	--
Trichlorofluoromethane	75-69-4	µg/Kg	NS	NS	NS	50	16	5	0.68	--	--
Vinyl chloride	75-01-4	µg/Kg	13,000	27,000	20	50	10	2	0.48	--	--

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Analyte	CAS number	Units	Restricted Use Soil Cleanup Objectives[2]			Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
			Commercial	Industrial	Protection of Groundwater	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
1,4-Dioxane (Method SW-846 8270D-Selective Ion Monitoring-SIM)											
1,4-Dioxane	123-91-1	µg/Kg	130,000	250,000	100	0.50	0.50	3.3	1.7	--	--

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			Commercial	Industrial	Protection of Groundwater	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Semi Volatile Organic Compounds (Method SW-846 8270D)											
1,1'-Biphenyl	92-52-4	µg/Kg	NS	NS	NS	330	11.5	67	4.6	--	--
1,2,4,5-Tetrachlorobenzene	95-94-3	µg/Kg	NS	NS	NS	330	10.3	170	8.5	--	--
2,2'-oxybis[1-chloropropane]	108-60-1	µg/Kg	NS	NS	NS	330	19.8	67	12	--	--
2,3,4,6-Tetrachlorophenol	58-90-2	µg/Kg	NS	NS	NS	330	22.4	170	22	--	--
2,4,5-Trichlorophenol	95-95-4	µg/Kg	NS	NS	100	330	33.7	170	25	--	--
2,4,6-Trichlorophenol	88-06-2	µg/Kg	NS	NS	NS	133	42.5	170	20	--	--
2,4-Dichlorophenol	120-83-2	µg/Kg	NS	NS	400	133	21.2	170	28	--	--
2,4-Dimethylphenol	105-67-9	µg/Kg	NS	NS	NS	330	39.5	170	59	--	--
2,4-Dinitrophenol	51-28-5	µg/Kg	NS	NS	200	266	163	170	130	--	--
2,4-Dinitrotoluene	121-14-2	µg/Kg	NS	NS	NS	67.0	35.6	33	10	--	--
2,6-Dinitrotoluene	606-20-2	µg/Kg	NS	NS	1,000	67.0	23.9	33	17	--	--
2-Chloronaphthalene	91-58-7	µg/Kg	NS	NS	NS	330	15.3	67	7.9	--	--
2-Chlorophenol	95-57-8	µg/Kg	NS	NS	NS	330	11.8	67	16	--	--
2-Methylnaphthalene	91-57-6	µg/Kg	NS	NS	36,400	330	9.25	33	7.5	--	--
2-Methylphenol	95-48-7	µg/Kg	500,000	1,000,000	330	330	12.4	67	21	--	--
2-Nitroaniline	88-74-4	µg/Kg	NS	NS	400	330	25.2	170	7.9	--	--
2-Nitrophenol	88-75-5	µg/Kg	NS	NS	300	330	33.1	170	22	--	--
3,3'-Dichlorobenzidine	91-94-1	µg/Kg	NS	NS	NS	133	50.0	67	28	--	--
3-Nitroaniline	99-09-2	µg/Kg	NS	NS	500	330	78.5	170	8.3	--	--
4,6-Dinitro-2-methylphenol	534-52-1	µg/Kg	NS	NS	NS	266	135	170	36	--	--
4-Bromophenyl phenyl ether	101-55-3	µg/Kg	NS	NS	NS	330	13.1	67	13	--	--
4-Chloro-3-methylphenol	59-50-7	µg/Kg	NS	NS	NS	330	18.6	170	20	--	--
4-Chloroaniline	106-47-8	µg/Kg	NS	NS	220	330	58.7	170	12	--	--
4-Chlorophenyl phenyl ether	7005-72-3	µg/Kg	NS	NS	NS	330	11.7	67	11	--	--
4-Methylphenol	106-44-5	µg/Kg	500,000	1,000,000	330	330	20.7	67	27	--	--
4-Nitroaniline	100-01-6	µg/Kg	NS	NS	NS	330	38.0	170	8.6	--	--
4-Nitrophenol	100-02-7	µg/Kg	NS	NS	100	670	53.9	330	89	--	--
Acenaphthene	83-32-9	µg/Kg	500,000	1,000,000	98,000	330	9.43	33	11	--	--
Acenaphthylene	208-96-8	µg/Kg	500,000	1,000,000	107,000	330	9.46	33	17	--	--
Acetophenone	98-86-2	µg/Kg	NS	NS	NS	330	16.2	170	7.2	--	--
Anthracene	120-12-7	µg/Kg	500,000	1,000,000	1,000,000	330	10.1	33	20	--	--
Atrazine	1912-24-9	µg/Kg	NS	NS	NS	133	19.5	67	14	--	--
Benzaldehyde	100-52-7	µg/Kg	NS	NS	NS	330	54.7	170	8.3	--	--
Benzo[a]anthracene	56-55-3	µg/Kg	5,600	11,000	1,000	33.0	24.9	33	9.4	--	--
Benzo[a]pyrene	50-32-8	µg/Kg	1,000	1,100	22,000	33.0	8.81	33	15	--	--
Benzo[b]fluoranthene	205-99-2	µg/Kg	5,600	11,000	1,700	33.0	8.56	33	15	--	--
Benzo[g,h,i]perylene	191-24-2	µg/Kg	500,000	1,000,000	10,000,000	330	9.76	33	17	--	--
Benzo[k]fluoranthene	207-08-9	µg/Kg	56,000	110,000	1,700	33.0	6.49	33	16	--	--
Bis(2-chloroethoxy)methane	111-91-1	µg/Kg	NS	NS	NS	330	25.8	67	7.1	--	--
Bis(2-chloroethyl)ether	111-44-4	µg/Kg	NS	NS	NS	33.0	11.5	67	14	--	--
Bis(2-ethylhexyl) phthalate	117-81-7	µg/Kg	NS	NS	435,000	330	17.5	67	7.8	--	--
Butyl benzyl phthalate	85-68-7	µg/Kg	NS	NS	122,000	330	15.5	67	8.1	--	--
Caprolactam	105-60-2	µg/Kg	NS	NS	NS	330	51.5	67	13	--	--
Carbazole	86-74-8	µg/Kg	NS	NS	NS	330	12.6	67	4.8	--	--
Chrysene	218-01-9	µg/Kg	56,000	110,000	1,000	330	13.9	33	10	--	--
Dibenz(a,h)anthracene	53-70-3	µg/Kg	560	1,100	1,000,000	33.0	14.3	33	15	--	--
Dibenzofuran	132-64-9	µg/Kg	350,000	1,000,000	210,000	330	11.0	67	14	--	--
Diethyl phthalate	84-66-2	µg/Kg	NS	NS	7,100	330	10.7	67	7.1	--	--
Dimethyl phthalate	131-11-3	µg/Kg	NS	NS	27,000	330	75.2	67	5.9	--	--
Di-n-butyl phthalate	84-74-2	µg/Kg	NS	NS	8,100	330	12.5	67	5.4	--	--
Di-n-octyl phthalate	117-84-0	µg/Kg	NS	NS	120,000	330	17.5	67	8.3	--	--
Fluoranthene	206-44-0	µg/Kg	500,000	1,000,000	1,000,000	330	11.6	33	15	--	--
Fluorene	86-73-7	µg/Kg	500,000	1,000,000	386,000	330	9.68	33	15	--	--
Hexachlorobenzene	118-74-1	µg/Kg	NS	NS	1,400	33.0	15.7	67	8.4	--	--
Hexachlorobutadiene	87-68-3	µg/Kg	NS	NS	NS	67.0	7.04	33	13	--	--
Hexachlorocyclopentadiene	77-47-4	µg/Kg	NS	NS	NS	330	29.0	330	13	--	--
Hexachloroethane	67-72-1	µg/Kg	NS	NS	NS	33.0	11.4	170	16	--	--
Indeno[1,2,3-cd]pyrene	193-39-5	µg/Kg	5,600	11,000	8,200	33.0	12.9	33	16	--	--
Isophorone	78-59-1	µg/Kg	NS	NS	4,400	133	95.6	67	7.1	--	--
Naphthalene	91-20-3	µg/Kg	500,000	1,000,000	12,000	330	5.72	33	9.4	--	--
Nitrobenzene	98-95-3	µg/Kg	69,000	140,000	170	33.0	18.3	67	13	--	--
N-Nitrosodi-n-propylamine	621-64-7	µg/Kg	NS	NS	NS	33.0	24.0	67	9.6	--	--
N-Nitrosodiphenylamine	86-30-6	µg/Kg	NS	NS	NS	330	27.2	170	12	--	--
Pentachlorophenol	87-86-5	µg/Kg	6,700	55,000	800	266	67.8	130	31	--	--
Phenanthrene	85-01-8	µg/Kg	500,000	1,000,000	1,000,000	330	13.5	33	11	--	--
Phenol	108-95-2	µg/Kg	500,000	1,000,000	330	330	12.2	67	17	--	--
Pyrene	129-00-0	µg/Kg	500,000	1,000,000	1,000,000	330	8.23	33	11	--	--

Table 1a
Parameters, Methods, and Target Reporting Limits
Quality Assurance Project Plan
Long Island MacArthur Airport
Ronkonkoma, New York

Analyte	CAS number	Units	Restricted Use Soil Cleanup Objectives[2]			Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
			Commercial	Industrial	Protection of Groundwater	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Metals/Mercury/Hexavlent Chromium (Methods SW-846 6010D/6020B/7471B/7196A)											
Silver	7440-22-4	mg/Kg	1,500	6,800	8.3	0.400	0.0890	0.5	0.17	--	--
Aluminum	7429-90-5	mg/Kg	NS	NS	NS	20.0	5.49	50	8.06	--	--
Arsenic	7440-38-2	mg/Kg	16	16	16	1.00	0.103	2	0.28	--	--
Barium	7440-39-3	mg/Kg	400	10,000	820	2.00	0.145	20	1.92	--	--
Beryllium	7440-41-7	mg/Kg	590	2,700	47	0.400	0.0570	0.2	0.08	--	--
Calcium	7440-70-2	mg/Kg	NS	NS	NS	100	40.7	500	20.8	--	--
Cadmium	7440-43-9	mg/Kg	9.3	60	7.5	1.00	0.113	0.5	0.07	--	--
Cobalt	7440-48-4	mg/Kg	NS	NS	NS	2.00	0.148	5	0.28	--	--
Chromium	7440-47-3	mg/Kg	1,500	6,800	NS	2.00	0.908	1	0.37	--	--
Copper	7440-50-8	mg/Kg	270	10,000	1,720	2.00	0.368	2.5	0.84	--	--
Iron	7439-89-6	mg/Kg	NS	NS	NS	60.0	20.2	50	19.2	--	--
Potassium	7440-09-7	mg/Kg	NS	NS	NS	100	16.2	1000	31.78	--	--
Magnesium	7439-95-4	mg/Kg	NS	NS	NS	100	10.2	500	13.67	--	--
Manganese	7439-96-5	mg/Kg	10,000	10,000	2,000	4.00	0.403	1.5	0.41	--	--
Sodium	7440-23-5	mg/Kg	NS	NS	NS	100	45.7	1000	77.81	--	--
Nickel	7440-02-0	mg/Kg	310	10,000	130	2.00	0.470	4	0.35	--	--
Lead	7439-92-1	mg/Kg	1,000	3,900	450	0.600	0.200	2	0.41	--	--
Antimony	7440-36-0	mg/Kg	NS	NS	NS	1.00	0.146	2	0.41	--	--
Selenium	7782-49-2	mg/Kg	1,500	6,800	4	1.25	0.128	2	0.65	--	--
Thallium	7440-28-0	mg/Kg	NS	NS	NS	0.400	0.0410	1	0.58	--	--
Vanadium	7440-62-2	mg/Kg	NS	NS	NS	2.00	0.206	5	0.19	--	--
Zinc	7440-66-6	mg/Kg	10,000	10,000	2,480	8.00	3.05	5	2.3	--	--
Mercury	7439-97-6	mg/Kg	2.8	5.7	0.73	0.0170	0.006	0.033	0.0145	--	--
Hexavalent Chromium	18540-29-9	mg/Kg	400	800	19	2	0.848	0.4	0.317	--	--

Analyte	CAS number	Units	Restricted Use Soil Cleanup Objectives[2]			Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
			Commercial	Industrial	Protection of Groundwater	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Polychlorinated Biphenyls (Method SW-846 8082B)											
Aroclor 1016	12674-11-2	µg/Kg	--	--	--	67	17.8	34	16	--	--
Aroclor 1221	11104-28-2	µg/Kg	--	--	--	67	17.8	34	21	--	--
Aroclor 1232	11141-16-5	µg/Kg	--	--	--	67	17.8	34	21	--	--
Aroclor 1242	53469-21-9	µg/Kg	--	--	--	67	17.8	34	14	--	--
Aroclor 1248	12672-29-6	µg/Kg	--	--	--	67	17.8	34	30	--	--
Aroclor 1254	11097-69-1	µg/Kg	--	--	--	67	17.8	34	18	--	--
Aroclor 1260	11096-82-5	µg/Kg	--	--	--	67	17.8	34	14	--	--
Arcolor 1262	37324-23-5	µg/Kg	--	--	--	67	17.8	34	29	--	--
Aroclor 1268	37324-23-5	µg/Kg	--	--	--	67	17.8	34	22	--	--
Total PCBs	1336-36-3	µg/Kg	1,000	25,000	3,200	67	17.8	37	30	--	--

Table 1a
Parameters, Methods, and Target Reporting Limits
Quality Assurance Project Plan
Long Island MacArthur Airport
Ronkonkoma, New York

ARCADIS Design & Consultancy for natural and built assets

Analyte	CAS number	Units	Restricted Use Soil Cleanup Objectives[2]			Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
			Commercial	Industrial	Protection of Groundwater	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Pesticides (Method SW-846 8081B)											
4,4'-DDD	72-54-8	µg/Kg	92,000	180,000	14,000	6.70	1.14	0.67	0.62	--	--
4,4'-DDE	72-55-9	µg/Kg	62,000	120,000	17,000	6.70	0.790	0.67	0.59	--	--
4,4'-DDT	50-29-3	µg/Kg	47,000	94,000	136,000	6.70	1.23	0.67	0.59	--	--
Aldrin	309-00-2	µg/Kg	680	1,400	190	6.70	1.01	0.67	0.55	--	--
alpha-BHC	319-84-6	µg/Kg	3,400	6,800	20	2.00	0.680	0.67	0.54	--	--
beta-BHC	319-85-7	µg/Kg	3,000	14,000	90	2.00	0.750	0.67	0.61	--	--
Chlordane (technical)	12789-03-6	µg/Kg	NS	NS	NS	67.0	16.2	0.67	0.49	--	--
delta-BHC	319-86-8	µg/Kg	500,000	1,000,000	250	2.00	0.410	0.67	0.64	--	--
Dieldrin	60-57-1	µg/Kg	1,400	2,800	100	2.00	0.870	0.67	0.46	--	--
Endosulfan I	959-98-8	µg/Kg	200,000	920,000	102,000	6.70	1.02	0.67	0.39	--	--
Endosulfan II	33213-65-9	µg/Kg	200,000	920,000	102,000	6.70	1.72	0.67	0.42	--	--
Endosulfan sulfate	1031-07-8	µg/Kg	200,000	920,000	1,000,000	6.70	0.840	0.67	0.52	--	--
Endrin	72-20-8	µg/Kg	89,000	410,000	60	6.70	0.960	0.67	0.52	--	--
Endrin aldehyde	7421-93-4	µg/Kg	NS	NS	NS	6.70	1.58	0.67	0.38	--	--
Endrin ketone	53494-70-5	µg/Kg	NS	NS	NS	6.70	1.30	0.67	0.48	--	--
gamma-BHC (Lindane)	58-89-9	µg/Kg	9,200	23,000	100	2.00	0.620	0.67	0.49	--	--
Heptachlor	76-44-8	µg/Kg	15,000	29,000	380	6.70	0.790	0.67	0.58	--	--
Heptachlor epoxide	1024-57-3	µg/Kg	NS	NS	20	6.70	1.00	0.67	0.47	--	--
Methoxychlor	72-43-5	µg/Kg	NS	NS	900,000	6.70	1.53	1.3	0.53	--	--
Toxaphene	8001-35-2	µg/Kg	NS	NS	NS	67.0	24.2	17	16	--	--

Analyte	CAS number	Units	Restricted Use Soil Cleanup Objectives[2]			Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
			Commercial	Industrial	Protection of Groundwater	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Herbicides (Method SW-846 8151A)											
2,4-D	94-75-7	µg/Kg	NS	NS	500	33.3	12.1	17	9	--	--
Silvex (2,4,5-TP)	93-72-1	µg/Kg	500,000	1,000,000	3,800	33.3	3.47	3.3	1.9	--	--
2,4,5-T	93-76-5	µg/Kg	NS	NS	1,900	33.3	7.08	3.3	1.6	--	--

Notes:

1. NYSDEC, Sampling, Analysis, and Assessment of PFAS Under NYSDEC's Part 375 Remedial Programs, April 2023. "NS" indicates there is no standard for the listed analyte.
2. NYSDEC, 6 NYCRR Part 375, Table 375-6.8(b): Restricted Use Soil Cleanp Objectives (SCO). "NS" indicates that there is no standard for the listed analyte.
3. Concentrations detected less than the RL but greater than the MDL must be reported with the appropriate qualifier. Not detected results will be reported at the laboratory reporting limit. Results will be reported on a dry weight basis and reporting limits adjusted accorgingly.
4. Laboratory reporting limits and method detection limits are updated on an annual basis. The limits listed are current as of the date of this QAPP. The laboratory will utilize their most current limits at the time of analysis.

Table 1b
Parameters, Methods, and Target Reporting Limits
Quality Assurance Project Plan
Long Island MacArthur Airport
Ronkonkoma, New York

Analyte	CAS number	Units	Groundwater Effluent Limitations[1,2]	Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
				Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Per- and Polyfluoroalkyl Substances (Method EPA 1633)									
Perfluorobutanoic acid (PFBA)	375-22-4	ng/L	NS	8	2	8	0.93	--	--
Perfluoropentanoic acid (PFPeA)	2706-90-3	ng/L	NS	4	1	4	0.73	--	--
Perfluorohexanoic acid (PFHxA)	307-24-4	ng/L	NS	2	0.5	2	0.29	--	--
Perfluoroheptanoic acid (PFHpA)	375-85-9	ng/L	NS	2	0.52	2	0.5	--	--
Perfluorooctanoic acid (PFOA)	335-67-1	ng/L	6.7	2	0.64	2	0.75	--	--
Perfluorononanoic acid (PFNA)	375-95-1	ng/L	NS	2	0.5	2	0.4	--	--
Perfluorodecanoic acid (PFDA)	335-76-2	ng/L	NS	2	0.5	2	0.62	--	--
Perfluoroundecanoic acid (PFUnA)	2058-94-8	ng/L	NS	2	0.5	2	0.55	--	--
Perfluorododecanoic acid (PFDoA)	307-55-1	ng/L	NS	2	0.5	2	0.77	--	--
Perfluorotridecanoic acid (PFTrDA)	72629-94-8	ng/L	NS	2	0.5	2	0.79	--	--
Perfluorotetradecanoic acid (PFTeDA)	376-06-7	ng/L	NS	2	0.5	2	0.76	--	--
Perfluorobutanesulfonic acid (PFBS)	375-73-5	ng/L	NS	2	0.3	2	0.66	--	--
Perfluoropentanesulfonic acid (PFPeS)	2706-91-4	ng/L	NS	2	0.5	2	1	--	--
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	ng/L	NS	2	0.57	2	1.1	--	--
Perfluoroheptanesulfonic acid (PFHpS)	375-92-8	ng/L	NS	2	0.4	2	0.98	--	--
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	ng/L	2.7	2	0.5	2	0.43	--	--
Perfluorononanesulfonic acid (PFNS)	68259-12-1	ng/L	NS	2	0.4	2	1.4	--	--
Perfluorododecanesulfonic acid (PFDoS)	79780-39-5	ng/L	NS	2	0.9	2	0.4	--	--
1H,1H,2H,2H-Perfluorohexane sulfonic acid (4:2 FTS)	757124-72-4	ng/L	NS	8	1.7	8	2.3	--	--
1H,1H,2H,2H-Perfluorooctane sulfonic acid (6:2 FTS)	27619-97-2	ng/L	NS	8	2.5	8	0.91	--	--
1H,1H,2H,2H-Perfluorodecane sulfonic acid (8:2 FTS)	39108-34-4	ng/L	NS	8	2.6	8	1.5	--	--
Perfluorooctanesulfonamide (PFOSA)	754-91-6	ng/L	NS	2	0.5	2	0.69	--	--
N-methylperfluorooctane sulfonamide (NMeFOSA)	31506-32-8	ng/L	NS	20	0.5	2	0.64	--	--
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	2355-31-9	ng/L	NS	4	1.2	2	0.51	--	--
N-ethylperfluorooctane sulfonamide (NEtFOSA)	4151-50-2	ng/L	NS	2	0.5	2	0.53	--	--
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	2991-50-6	ng/L	NS	2	0.7	2	0.91	--	--
N-methylperfluorooctane sulfonamidoethanol (NMeFOSE)	24448-09-7	ng/L	NS	20	5	20	2.8	--	--
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	1691-99-2	ng/L	NS	20	5	20	3.8	--	--
Hexafluoropropylene Oxide Dimer Acid (HFPO-DA)	13252-13-6	ng/L	NS	8	2	8	1.8	--	--
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	919005-14-4	ng/L	NS	8	1.5	8	1.2	--	--
Perfluoro-4-methoxybutanoic acid (PFMBA)	863090-89-5	ng/L	NS	4	1	4	0.56	--	--
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	151772-58-6	ng/L	NS	4	1	4	1.3	--	--
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	756426-58-1	ng/L	NS	8	1	8	2.4	--	--
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	763051-92-9	ng/L	NS	8	2	8	1.8	--	--
Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)	113507-82-7	ng/L	NS	4	0.5	4	0.27	--	--
3-Perfluoropropylpropanoic acid (3:3 FTCA)	356-02-5	ng/L	NS	10	1.5	10	2.4	--	--
3-Perfluoropentylpropanoic acid (5:3 FTCA)	914637-49-3	ng/L	NS	50	10	50	7.1	--	--
3-Perfluoroheptylpropanoic acid (7:3 FTCA)	812-70-4	ng/L	NS	50	10	50	6.2	--	--
Perfluorodecanesulfonic acid (PFDS)	335-77-3	ng/L	NS	2	0.5	2	1.1	--	--
Perfluoro-3-methoxypropanoic acid (PFMPA)	377-73-1	ng/L	NS	4	0.5	4	0.3	--	--

Table 1b
Parameters, Methods, and Target Reporting Limits
Quality Assurance Project Plan
Long Island MacArthur Airport
Ronkonkoma, New York

Analyte	CAS number	Units	Groundwater Effluent Limitations[1,2]	Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
				Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Per- and Polyfluoroalkyl Substances (Method ASTM D8421)									
Perfluorobutanoic acid (PFBA)	375-22-4	ng/L	NS	--	--	80	36	50	8.3
Perfluoropentanoic acid (PFPeA)	2706-90-3	ng/L	NS	--	--	40	8	50	14
Perfluorohexanoic acid (PFHxA)	307-24-4	ng/L	NS	--	--	20	6	10	1.4
Perfluoroheptanoic acid (PFHpA)	375-85-9	ng/L	NS	--	--	20	8	10	3.2
Perfluorooctanoic acid (PFOA)	335-67-1	ng/L	6.7	--	--	20	6	10	4.1
Perfluorononanoic acid (PFNA)	375-95-1	ng/L	NS	--	--	20	10	10	2.2
Perfluorodecanoic acid (PFDA)	335-76-2	ng/L	NS	--	--	20	6	10	2.2
Perfluoroundecanoic acid (PFUnA)	2058-94-8	ng/L	NS	--	--	20	6	10	1.4
Perfluorododecanoic acid (PFDoA)	307-55-1	ng/L	NS	--	--	20	10	10	2
Perfluorotridecanoic acid (PFTrDA)	72629-94-8	ng/L	NS	--	--	20	18	10	5.8
Perfluorotetradecanoic acid (PFTeDA)	376-06-7	ng/L	NS	--	--	20	10	50	50
Perfluorobutanesulfonic acid (PFBS)	375-73-5	ng/L	NS	--	--	20	12	10	3.5
Perfluoropentanesulfonic acid (PFPeS)	2706-91-4	ng/L	NS	--	--	20	18	10	2.9
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	ng/L	NS	--	--	20	14	10	1.6
Perfluoroheptanesulfonic acid (PFHpS)	375-92-8	ng/L	NS	--	--	20	20	10	1.6
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	ng/L	2.7	--	--	20	18	10	1.6
Perfluorononanesulfonic acid (PFNS)	68259-12-1	ng/L	NS	--	--	20	16	10	1.9
Perfluorododecanesulfonic acid (PFDoS)	79780-39-5	ng/L	NS	--	--	40	38	50	50
1H,1H,2H,2H-Perfluorohexane sulfonic acid (4:2 FTS)	757124-72-4	ng/L	NS	--	--	80	20	10	1.9
1H,1H,2H,2H-Perfluorooctane sulfonic acid (6:2 FTS)	27619-97-2	ng/L	NS	--	--	80	20	20	4.4
1H,1H,2H,2H-Perfluorodecane sulfonic acid (8:2 FTS)	39108-34-4	ng/L	NS	--	--	80	34	10	5.2
Perfluorooctanesulfonamide (PFOSA)	754-91-6	ng/L	NS	--	--	20	12	10	1.6
N-methylperfluorooctane sulfonamide (NMeFOSA)	31506-32-8	ng/L	NS	--	--	40	28	10	2.1
N-methylperfluorooctanesulfonamidoacetic acid (NMeFOSAA)	2355-31-9	ng/L	NS	--	--	20	12	10	2.7
N-ethylperfluorooctane sulfonamide (NEtFOSA)	4151-50-2	ng/L	NS	--	--	40	34	10	2.6
N-ethylperfluorooctanesulfonamidoacetic acid (NEtFOSAA)	2991-50-6	ng/L	NS	--	--	20	18	10	3.5
N-methylperfluorooctane sulfonamidoethanol (NMeFOSE)	24448-09-7	ng/L	NS	--	--	200	62	10	4.5
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	1691-99-2	ng/L	NS	--	--	200	78	10	2.6
Hexafluoropropylene Oxide Dimer Acid (HFPO-DA)	13252-13-6	ng/L	NS	--	--	80	74	10	2.4
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	919005-14-4	ng/L	NS	--	--	80	26	10	1.7
Perfluoro-4-methoxybutanoic acid (PFMBA)	863090-89-5	ng/L	NS	--	--	160	12	10	2
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	151772-58-6	ng/L	NS	--	--	160	12	10	1.8
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	756426-58-1	ng/L	NS	--	--	80	36	10	1.8
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	763051-92-9	ng/L	NS	--	--	80	22	10	3
Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)	113507-82-7	ng/L	NS	--	--	160	38	10	1
3-Perfluoropropylpropanoic acid (3:3 FTCA)	356-02-5	ng/L	NS	--	--	100	38	40	5
3-Perfluoropentylpropanoic acid (5:3 FTCA)	914637-49-3	ng/L	NS	--	--	500	68	40	4.9
3-Perfluoroheptylpropanoic acid (7:3 FTCA)	812-70-4	ng/L	NS	--	--	500	110	40	2.7
Perfluorodecanesulfonic acid (PFDS)	335-77-3	ng/L	NS	--	--	20	20	10	2.6
Perfluoro-3-methoxypropanoic acid (PFMPA)	377-73-1	ng/L	NS	--	--	160	64	10	1.4

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Volatile Organic Compounds (Method SW-846 8260D)									
1,1,1-Trichloroethane	71-55-6	µg/L	5	1.00	0.238	1	0.54	--	--
1,1,2,2-Tetrachloroethane	79-34-5	µg/L	5	1.00	0.367	1	0.65	--	--
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	µg/L	5	1.00	0.311	5	0.58	--	--
1,1,2-Trichloroethane	79-00-5	µg/L	1	1.00	0.204	1	0.53	--	--
1,1-Dichloroethane	75-34-3	µg/L	5	1.00	0.264	1	0.57	--	--
1,1-Dichloroethene	75-35-4	µg/L	5	1.00	0.264	1	0.59	--	--
1,2,3-Trichlorobenzene	87-61-6	µg/L	5	1.00	0.357	1	0.5	--	--
1,2,4-Trichlorobenzene	120-82-1	µg/L	5	1.00	0.365	--	--	--	--
1,2-Dichloropropane	78-87-5	µg/L	1	1.00	0.353	1	0.51	--	--
1,3-Dichlorobenzene	541-73-1	µg/L	3	1.00	0.342	1	0.54	--	--
1,4-Dichlorobenzene	106-46-7	µg/L	3	1.00	0.334	1	0.51	--	--
2-Butanone (MEK)	78-93-3	µg/L	50	5.00	1.85	10	2.7	--	--
2-Hexanone	591-78-6	µg/L	50	5.00	1.14	5	4.8	--	--
4-Methyl-2-pentanone (MIBK)	108-10-1	µg/L	NS	5.00	1.30	5	4.9	--	--
Acetone	67-64-1	µg/L	50	5.00	4.42	10	3.1	--	--
Benzene	71-43-2	µg/L	1	1.00	0.203	0.5	0.43	--	--
Bromoform	75-25-2	µg/L	50	1.00	0.536	1	0.63	--	--
Bromomethane	74-83-9	µg/L	5	1.00	0.550	2	1.6	--	--
Carbon disulfide	75-15-0	µg/L	NS	1.00	0.821	2	1.8	--	--
Carbon tetrachloride	56-23-5	µg/L	5	1.00	0.208	1	0.55	--	--
Chlorobenzene	108-90-7	µg/L	5	1.00	0.377	1	0.56	--	--
Chlorobromomethane	74-97-5	µg/L	5	1.00	0.412	1	0.48	--	--
Chlorodibromomethane	124-48-1	µg/L	50	1.00	0.281	1	0.56	--	--
Chloroethane	75-00-3	µg/L	5	1.00	0.320	1	0.73	--	--
Chloroform	67-66-3	µg/L	7	1.00	0.326	1	0.5	--	--
Chloromethane	74-87-3	µg/L	5	1.00	0.402	1	0.76	--	--
cis-1,2-Dichloroethene	156-59-2	µg/L	5	1.00	0.219	1	0.51	--	--
cis-1,3-Dichloropropene	10061-01-5	µg/L	0.4	1.00	0.222	1	0.47	--	--
trans-1,3-Dichloropropene	10061-02-6	µg/L		1.00	0.223	1	0.43	--	--
Cyclohexane	110-82-7	µg/L	NS	1.00	0.321	5	0.78	--	--
Dichlorobromomethane	75-27-4	µg/L	50	1.00	0.343	1	0.45	--	--
Dichlorodifluoromethane	75-71-8	µg/L	5	1.00	0.311	2	0.56	--	--
Ethylbenzene	100-41-4	µg/L	5	1.00	0.298	1	0.6	--	--
Ethylene Dibromide	106-93-4	µg/L	0.0006	1.00	0.498	1	0.48	--	--
Isopropylbenzene	98-82-8	µg/L	5	1.00	0.336	1	0.65	--	--
Methyl acetate	79-20-9	µg/L	NS	5.00	0.785	5	0.8	--	--
Methyl tert-butyl ether	1634-04-4	µg/L	NS	1.00	0.216	1	0.51	--	--
Methylcyclohexane	108-87-2	µg/L	NS	1.00	0.707	5	0.6	--	--
Methylene Chloride	75-09-2	µg/L	5	1.00	0.315	2	1	--	--
m-Xylene & p-Xylene	179601-23-1	µg/L	10	1.00	0.296	1	0.78	--	--
o-Xylene	95-47-6	µg/L	5	1.00	0.361	1	0.59	--	--
Styrene	100-42-5	µg/L	930	1.00	0.415	1	0.49	--	--
Tetrachloroethene	127-18-4	µg/L	5	1.00	0.249	1	0.56	--	--
Toluene	108-88-3	µg/L	5	1.00	0.379	1	0.49	--	--
trans-1,2-Dichloroethene	156-60-5	µg/L	5	1.00	0.235	1	0.54	--	--
Trichloroethene	79-01-6	µg/L	5	1.00	0.314	1	0.53		
Trichlorofluoromethane	75-69-4	µg/L	5	1.00	0.320	2	0.4		
Vinyl chloride	75-01-4	µg/L	2	1.00	0.171	1	0.52	--	--
1,2-Dichloroethane	107-06-2	µg/L	0.6	1.00	0.430	1	0.6	--	--
1,2-Dichlorobenzene	95-50-1	µg/L	3	1.00	0.212	1	0.53	--	--
1,2-Dibromo-3-Chloropropane	96-12-8	µg/L	0.04	1.00	0.376	2	0.53	--	--

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1,4-Dioxane (Method SW-846 8270D-Selective Ion Monitoring-SIM)									
1,4-Dioxane	123-91-1	µg/L	0.35	0.200	0.072	0.1	0.05	--	--

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Semi Volatile Organic Compounds (Method SW-846 8270D)									
2,4-Dichlorophenol	120-83-2	µg/L		10.0	1.07	2	1.3	--	--
2,4-Dimethylphenol	105-67-9	µg/L		10.0	0.619	5	2.4	--	--
2,4-Dinitrophenol	51-28-5	µg/L	2	40.0	2.63	5	1.6	--	--
Pentachlorophenol	87-86-5	µg/L		20.0	1.45	4	1.4	--	--
Phenol	108-95-2	µg/L		10.0	0.292	2	0.39	--	--
1,1'-Biphenyl	92-52-4	µg/L	5	10.0	1.19	1	0.21	--	--
1,2,4,5-Tetrachlorobenzene	95-94-3	µg/L	5	10.0	1.24	2	0.37	--	--
2,2'-oxybis[1-chloropropane]	108-60-1	µg/L	5	10.0	0.629	2	0.4	--	--
2,3,4,6-Tetrachlorophenol	58-90-2	µg/L	NS	10.0	0.746	5	1.5	--	--
2,4,5-Trichlorophenol	95-95-4	µg/L	NS	10.0	0.880	5	1.3	--	--
2,4,6-Trichlorophenol	88-06-2	µg/L	NS	10.0	0.857	5	0.92	--	--
2,4-Dinitrotoluene	121-14-2	µg/L	5	10.0	0.997	1	0.55	--	--
2,6-Dinitrotoluene	606-20-2	µg/L	5	2.00	0.826	1	0.48	--	--
2-Chloronaphthalene	91-58-7	µg/L	10	10.0	1.18	2	0.24	--	--
2-Chlorophenol	95-57-8	µg/L	NS	10.0	0.377	5	0.82	--	--
2-Methylnaphthalene	91-57-6	µg/L	NS	10.0	0.527	1	0.21	--	--
2-Methylphenol	95-48-7	µg/L	NS	10.0	0.671	2	0.89	--	--
2-Nitroaniline	88-74-4	µg/L	5	10.0	0.474	5	0.28	--	--
2-Nitrophenol	88-75-5	µg/L	NS	10.0	0.747	5	0.96	--	--
3,3'-Dichlorobenzidine	91-94-1	µg/L	5	10.0	1.43	2	0.51	--	--
3-Nitroaniline	99-09-2	µg/L	5	10.0	1.94	5	0.39	--	--
4,6-Dinitro-2-methylphenol	534-52-1	µg/L	NS	20.0	2.99	5	1.3	--	--
4-Bromophenyl phenyl ether	101-55-3	µg/L	NS	10.0	0.745	2	0.4	--	--
4-Chloro-3-methylphenol	59-50-7	µg/L	NS	10.0	0.575	5	0.89	--	--
4-Chloroaniline	106-47-8	µg/L	5	10.0	1.88	5	0.34	--	--
4-Chlorophenyl phenyl ether	7005-72-3	µg/L	NS	10.0	1.28	2	0.37	--	--
4-Methylphenol	106-44-5	µg/L	NS	10.0	0.651	2	0.88	--	--
4-Nitroaniline	100-01-6	µg/L	5	10.0	1.22	5	0.44	--	--
4-Nitrophenol	100-02-7	µg/L	NS	20.0	3.98	10	1.2	--	--
Acenaphthene	83-32-9	µg/L	20	10.0	1.08	1	0.19	--	--
Acenaphthylene	208-96-8	µg/L	NS	10.0	0.823	1	0.14	--	--
Acetophenone	98-86-2	µg/L	NS	10.0	2.33	2	0.21	--	--
Anthracene	120-12-7	µg/L	50	10.0	1.30	1	0.21	--	--
Atrazine	1912-24-9	µg/L	7.5	2.00	1.35	2	0.45	--	--
Benzaldehyde	100-52-7	µg/L	NS	10.0	2.10	5	0.29	--	--
Benzo[a]anthracene	56-55-3	µg/L	0.002	1.00	0.592	1	0.2	--	--
Benzo[a]pyrene	50-32-8	µg/L	ND	1.00	0.405	1	0.21	--	--
Benzo[b]fluoranthene	205-99-2	µg/L	0.002	2.00	0.676	1	0.21	--	--
Benzo[g,h,i]perylene	191-24-2	µg/L	NS	10.0	0.702	1	0.34	--	--
Benzo[k]fluoranthene	207-08-9	µg/L	0.002	1.00	0.674	1	0.21	--	--
Bis(2-chloroethoxy)methane	111-91-1	µg/L	5	10.0	0.589	2	0.28	--	--
Bis(2-chloroethyl)ether	111-44-4	µg/L	1	1.00	0.633	2	0.25	--	--
Bis(2-ethylhexyl) phthalate	117-81-7	µg/L	5	2.00	0.804	2	1.7	--	--
Butyl benzyl phthalate	85-68-7	µg/L	50	10.0	0.854	2	0.46	--	--
Caprolactam	105-60-2	µg/L	NS	10.0	2.24	2	0.65	--	--
Carbazole	86-74-8	µg/L	NS	10.0	0.679	1	0.23	--	--
Chrysene	218-01-9	µg/L	0.002	2.00	0.907	1	0.18	--	--
Dibenz(a,h)anthracene	53-70-3	µg/L	NS	1.00	0.720	1	0.33	--	--
Dibenzofuran	132-64-9	µg/L	NS	10.0	1.10	5	0.22	--	--
Diethyl phthalate	84-66-2	µg/L	50	10.0	0.976	2	0.26	--	--
Dimethyl phthalate	131-11-3	µg/L	50	10.0	0.766	2	0.22	--	--
Di-n-butyl phthalate	84-74-2	µg/L	50	10.0	0.840	2	0.5	--	--
Di-n-octyl phthalate	117-84-0	µg/L	50	10.0	0.749	2	0.23	--	--
Fluoranthene	206-44-0	µg/L	50	10.0	0.842	1	0.17	--	--
Fluorene	86-73-7	µg/L	50	10.0	0.912	1	0.17	--	--
Hexachlorobenzene	118-74-1	µg/L	0.04	1.00	0.396	1	0.33	--	--
Hexachlorobutadiene	87-68-3	µg/L	0.5	1.00	0.780	1	0.49	--	--
Hexachlorocyclopentadiene	77-47-4	µg/L	5	10.0	3.64	10	2.8	--	--
Hexachloroethane	67-72-1	µg/L	5	2.00	0.803	2	0.39	--	--
Indeno[1,2,3-cd]pyrene	193-39-5	µg/L	0.002	2.00	0.939	1	0.33	--	--
Isophorone	78-59-1	µg/L	50	10.0	0.798	2	0.28	--	--
Naphthalene	91-20-3	µg/L	10	2.00	0.541	1	0.23	--	--
Nitrobenzene	98-95-3	µg/L	0.4	1.00	0.567	2	0.64	--	--
N-Nitrosodi-n-propylamine	621-64-7	µg/L	NS	1.00	0.430	2	0.48	--	--
N-Nitrosodiphenylamine	86-30-6	µg/L	50	10.0	0.891	5	0.22	--	--
Phenanthrene	85-01-8	µg/L	50	10.0	1.28	1	0.18	--	--
Pyrene	129-00-0	µg/L	50	10.0	1.64	1	0.22	--	--

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Metals/Mercury/Hexavlent Chromium (Methods SW-846 6010D/6020B/7470A/7196A)									
Silver	7440-22-4	µg/L	100	2.00	0.290	10	6.07	--	--
Aluminum	7429-90-5	µg/L	2,000	40.0	19.5	200	148	--	--
Arsenic	7440-38-2	µg/L	50	2.00	0.887	8	2.76	--	--
Barium	7440-39-3	µg/L	2,000	4.00	0.913	200	97.18	--	--
Beryllium	7440-41-7	µg/L	3	0.800	0.130	1	0.5	--	--
Calcium	7440-70-2	µg/L	NS	500	53.6	5000	99.2	--	--
Cadmium	7440-43-9	µg/L	10.0	2.00	0.386	3	1.04	--	--
Cobalt	7440-48-4	µg/L	NS	4.00	0.707	50	2.57	--	--
Chromium	7440-47-3	µg/L	100	4.00	2.50	10	1.99	--	--
Copper	7440-50-8	µg/L	1,000	4.00	2.45	10	5.85	--	--
Iron	7439-89-6	µg/L	600	120	58.2	100	32.11	--	--
Potassium	7440-09-7	µg/L	NS	200	112	10000	200	--	--
Magnesium	7439-95-4	µg/L	35,000	200	46.9	5000	139.77	--	--
Manganese	7439-96-5	µg/L	600	8.00	1.47	15	1.44	--	--
Sodium	7440-23-5	µg/L	NS	500	219	10000	568.6	--	--
Nickel	7440-02-0	µg/L	200	4.00	0.905	10	1.71	--	--
Lead	7439-92-1	µg/L	50	1.20	0.844	3	1.8	--	--
Antimony	7440-36-0	µg/L	6	2.00	0.757	6	4.67	--	--
Selenium	7782-49-2	µg/L	20	2.50	0.589	10	4.94	--	--
Thallium	7440-28-0	µg/L	0.5	0.800	0.208	10	1.76	--	--
Vanadium	7440-62-2	µg/L	NS	4.00	0.681	50	1.79	--	--
Zinc	7440-66-6	µg/L	5,000	16.0	6.52	20	6.9	--	--
Mercury	7439-97-6	µg/L	1.4	0.200	0.0910	0.2	0.0952	--	--
Hexavalent Chromium	18540-29-9	µg/L	100	10	8.14	10	2.6	--	--

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Polychlorinated Biphenyls (Method SW-846 8082B)									
Aroclor 1016	12674-11-2	µg/L	NS	0.400	0.119	0.25	0.098	--	--
Aroclor 1221	11104-28-2	µg/L	NS	0.400	0.119	0.25	0.21	--	--
Aroclor 1232	11141-16-5	µg/L	NS	0.400	0.119	0.25	0.13	--	--
Aroclor 1242	53469-21-9	µg/L	NS	0.400	0.119	0.25	0.23	--	--
Aroclor 1248	12672-29-6	µg/L	NS	0.400	0.119	0.25	0.22	--	--
Aroclor 1254	11097-69-1	µg/L	NS	0.400	0.107	0.25	0.21	--	--
Aroclor 1260	11096-82-5	µg/L	NS	0.400	0.107	0.25	0.21	--	--
Arcolor 1262	37324-23-5	µg/L	NS	0.400	0.107	0.25	0.087	--	--
Aroclor 1268	37324-23-5	µg/L	NS	0.400	0.107	0.25	0.23	--	--
Total PCBs	1336-36-3	µg/L	0.09	0.400	0.119	0.25	0.23	--	--

Table 1b
Parameters, Methods, and Target Reporting Limits
Quality Assurance Project Plan
Long Island MacArthur Airport
Ronkonkoma, New York

Analyte	CAS number	Units	Groundwater Effluent Limitations[1,2]	Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
				Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Pesticides (Method SW-846 8081B)									
4,4'-DDD	72-54-8	µg/L	0.3	0.0200	0.00600	0.005	0.0029	--	--
4,4'-DDE	72-55-9	µg/L	0.2	0.0200	0.00200	0.005	0.0025	--	--
4,4'-DDT	50-29-3	µg/L	0.2	0.0200	0.00400	0.005	0.0034	--	--
Aldrin	309-00-2	µg/L	ND	0.0200	0.00300	0.005	0.0026	--	--
alpha-BHC	319-84-6	µg/L	0.01	0.0200	0.00700	0.005	0.0026	--	--
beta-BHC	319-85-7	µg/L	0.04	0.0200	0.0150	0.005	0.004	--	--
Chlordane (technical)	12789-03-6	µg/L	0.05	0.500	0.0550	0.005	0.0025	--	--
delta-BHC	319-86-8	µg/L	0.04	0.0200	0.00500	0.005	0.0033	--	--
Dieldrin	60-57-1	µg/L	0.004	0.0200	0.00300	0.005	0.0038	--	--
Endosulfan I	959-98-8	µg/L	NS	0.0200	0.00200	0.005	0.0026	--	--
Endosulfan II	33213-65-9	µg/L	NS	0.0200	0.00400	0.005	0.0024	--	--
Endosulfan sulfate	1031-07-8	µg/L	NS	0.0200	0.00600	0.005	0.0027	--	--
Endrin	72-20-8	µg/L	ND	0.0200	0.00400	0.005	0.003	--	--
Endrin aldehyde	7421-93-4	µg/L	5	0.0200	0.00800	0.005	0.0034	--	--
Endrin ketone	53494-70-5	µg/L	5	0.0200	0.00800	0.005	0.0031	--	--
gamma-BHC (Lindane)	58-89-9	µg/L	0.05	0.0200	0.0120	0.005	0.003	--	--
Heptachlor	76-44-8	µg/L	0.04	0.0200	0.00300	0.005	0.0022	--	--
Heptachlor epoxide	1024-57-3	µg/L	0.03	0.0200	0.00500	0.005	0.003	--	--
Methoxychlor	72-43-5	µg/L	35	0.0200	0.00400	0.01	0.0034	--	--
Toxaphene	8001-35-2	µg/L	0.06	0.500	0.110	0.13	0.082	--	--

Analyte	CAS number	Units	Groundwater Effluent Limitations[1,2]	Eurofins Limits[3,4]		SGS Limits[3,4]		Pace Limits[3,4]	
				Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit	Laboratory Reporting Limit	Laboratory Method Detection Limit
Herbicides (Method SW-846 8151A)									
2,4-D	94-75-7	µg/L	50	1.20	0.130	0.5	0.24	--	--
Silvex (2,4,5-TP)	93-72-1	µg/L	0.26	1.20	0.110	01	0.063	--	--
2,4,5-T	93-76-5	µg/L	35	1.20	0.120	0.1	0.054	--	--

Notes:

1. NYSDEC, Sampling, Analysis, and Assessment of PFAS Under NYSDEC's Part 375 Remedial Programs, April 2023. "NS" indicates there is no standard for the listed analyte. Shaded cells indicate that the listed screening limit is less than the laboratory reporting limit and/or method detection limit.

2. NYSDEC, Division of Water Technical and Operational Guidance Series (1.1.1) Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations. "NS" indicates that there is no standard for the listed analyte. Shaded cells indicate that the screening limit is less than the laboratory reporting limit and/or method detection limit.
3. Concentrations detected less than the RL but greater than the MDL must be reported with the appropriate qualifier. Not detected results will be reported at the laboratory reporting limit.
4. Laboratory reporting limits and method detection limits are updated on an annual basis. The limits listed are current as of the date of this QAPP. The laboratory will utilize their most current limits at the time of analysis.

Table 2
Analytical Quality Control Limits[1]
Quality Assurance Project Plan
Long Island MacArthur Airport
Ronkonkoma, New York

ARCADIS Design & Consultancy for natural and built assets

	Accuracy - % Recovery[1,2]			Precision - RPD[1,2]			
Parameter	**Surrogate**	**MS/MSD**	**LCS**	**MS/MSD**	**LCS/LCSD**	**Lab Duplicate**	**Field Duplicate**
Groundwater							
PFAS	USEPA Method 1633 limits for EIS	--	USEPA Method 1633 limits	--	USEPA Method 1633 limits	--	30
1,4-Dioxane	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	30
VOCs	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	30
SVOCs	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	30
Metals	--	75-125	80-120	20	20	20	30
Merury	--	75-125	80-120	20	20	20	30
Hexavalent Chromium	--	75-125	80-120	20	20	20	30
PCBs	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	30
Pesticides	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	30
Herbicides	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	30
Soil							
PFAS	USEPA Method 1633 limits for EIS	--	USEPA Method 1633 limits	--	USEPA Method 1633 limits	--	50
1,4-Dioxane	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	50
VOCs	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	50
SVOCs	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	50
Metals	--	75-125	80-120	35	35	35	50
Mercury	--	75-125	80-120	35	35	35	50
Hexavalent Chromium	--	75-125	80-120	35	35	35	50
PCBs	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	50
Pesticides	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	50
Herbicides	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	Lab Established Limits	--	50

Notes:

1, For PFAS analysis, the laboratory will adhere to the limits provided in version of USEPA Method 1633 published at the time of sample analysis.

2. The QC limits for all other parameters will be based on the laboratory-generated limits at the time of analysis.

RPD Relative percent difference

PFAS Per- and polyfluoroalkyl substances

VOCs Volatile organic compounds

SVOCs Semi-volatile organic compounds

PCBs Polychlorinated Biphenyls

Table 3
Sample Quantities and Quality Control Frequencies
Quality Assurance Project Plan
Long Island MacArthur Airport
Ronkonkoma, New York

Parameter	Method	Estimated Environmental Sample Quantity*	Field QC Analyses						Laboratory QC Sample						Total
			Trip Blank		Rinse Blank		Field Duplicate		MS		MSD		Lab Duplicate		
			Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Groundwater															
PFAS	USEPA 1633	15	NA	--	1/day	TBD	1/20	2	NA	--	NA	--	NA	--	17
PFAS	ASTM D8421	150	NA	--	1/day	TBD	1/20	15	NA	--	NA	--	NA	--	165
VOCs	SW-846 8260C	0	1/cooler	--	1/day	TBD	1/20	0	1/20	0	1/20	0	NA	--	0
SVOCs, including 1,4-Dioxane	SW-846 8270E/8270E-SIM	0	NA	--	1/day	TBD	1/20	0	1/20	0	1/20	0	NA	--	0
Metals	SW-846 6010D	0	NA	--	1/day	TBD	1/20	0	1/20	0	1/20	0	1/20	0	0
Mercury	SW-846 7470A	0	NA	--	1/day	TBD	1/20	0	1/20	0	1/20	0	1/20	0	0
Hexavalent Chromium	SW-846 7196A	0	NA	--	1/day	TBD	1/20	0	1/20	0	1/20	0	1/20	0	0
PCBs	SW-846 8082A	0	NA	--	1/day	TBD	1/20	0	1/20	0	1/20	0	NA	--	0
Pesticides	SW-846 8081B	0	NA	--	1/day	TBD	1/20	0	1/20	0	1/20	0	NA	--	0
Herbicides	SW-846 8151A	0	NA	--	1/day	TBD	1/20	0	1/20	0	1/20	0	NA	--	0
Soil															
PFAS	USEPA 1633	10	NA	--	1/day	TBD	1/20	1	NA	--	NA	--	NA	--	11
PFAS	ASTM D8421	97	NA	--	1/day	TBD	1/20	10	NA	--	NA	--	NA	--	107
VOCs	SW-846 8260C	10	1/cooler	TBD	1/day	TBD	1/20	1	1/20	1	1/20	1	NA	--	13
SVOCs, including 1,4-Dioxane	SW-846 8270E/8270E-SIM	10	NA	--	1/day	TBD	1/20	1	1/20	1	1/20	1	NA	--	13
Metals	SW-846 6010D	10	NA	--	1/day	TBD	1/20	1	1/20	1	1/20	1	1/20	1	14
Mercury	SW-846 7471B	10	NA	--	1/day	TBD	1/20	1	1/20	1	1/20	1	1/20	1	14
Hexavalent Chromium	SW-846 7196A	10	NA	--	1/day	TBD	1/20	1	1/20	1	1/20	1	1/20	1	14
PCBs	SW-846 8082A	10	NA	--	1/day	TBD	1/20	1	1/20	1	1/20	1	NA	--	13
Pesticides	SW-846 8081B	10	NA	--	1/day	TBD	1/20	1	1/20	1	1/20	1	NA	--	13
Herbicides	SW-846 8151A	10	NA	--	1/day	TBD	1/20	1	1/20	1	1/20	1	NA	--	13

*Estimated only, total number of samples will vary depending on field conditions

Abbreviations and Acronyms
Freq = Frequency
MS = Matrix Spike
MSD = Matrix Spike Duplicate
NA = Not Applicable
No. = Number
PCBs = polychlorinated Biphenyls
PFAS = per- and polyfluoroalkyl substances
QC = Quality Control
SVOCs = Semivolatile Organic Compounds
TBD = To Be Determined
VOCs = Volatile Organic Compounds

Table 4
Sample Containers, Preservation, and Holding Times
Quality Assurance Project Plan
Long Island MacArthur Airport
Ronkonkoma, New York

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Parameter	Method[1]	Bottle Type[2]	Preservation	Holding Time[3]
Groundwater				
PFAS	USEPA 1633	1- 500 mL HDPE bottle with linerless HDPE or polypropylene caps	Cool to <6°C; samples may be stored at <-20ºC until extraction	28 days from colelction to extraction when stored <6ºC/90 days to extraction when stored <-20ºC, 28 days from extraction to analysis
PFAS	ASTM D8421	3- 15mL PP bottles	Cool to <6°C	28 days from collection to analysis
VOCs	SW-846 8260C	3 - 40 mL glass vials with Teflon®-lined lid	HCl to pH<2, no head space, Cool to <6°C	14 days from collection to analysis
SVOCs, including 1,4-Dioxane	SW-846 8270E/8270E-SIM	2- 250mL glass bottle with Teflon®-lined lid	Cool to <6°C	7 days from collection to extraction, 40 days from extraction to analysis
Metals	SW-846 6010D	1- 250 mL Plastic or glass bottle	HNO3 to pH<2, Cool to <6°C	180 days from collection to analysis (inclusive of digestion and analysis)
Mercury	SW-846 7470A	1- 250 mL Plastic or glass bottle	HNO3 to pH<2, Cool to <6°C	28 days from collection to analysis (inclusive of digestion and analysis)
Hexavalent Chromium	SW-846 7196A	1- 250mL HDPE bottle	Cool to <6°C	24 hours from collection to analysis
PCBs	SW-846 8082A	2- 250mL amber glass bottle	Cool to <6°C	7 days from collection to extraction, 40 days from extraction to analysis
Pesticides	SW-846 8081B	2- 250mL amber glass bottle	Cool to <6°C	7 days from collection to extraction, 40 days from extraction to analysis
Herbicides	SW-846 8151A	2- 1L amber glass bottle	Cool to <6°C	7 days from collection to extraction, 40 days from extraction to analysis
Soil				
PFAS	USEPA 1633	1- 4oz PP specimen container with polyethylene screw cap	Cool to <6°C; samples may be stored at <-20ºC until extraction	90 days from collection to extraction, 28 days from extraction to analysis
PFAS	ASTM D8421	1- 3oz or 90mL linerless polypropylene staight sides bottle	Cool to <6 °C	28 days from collection to analysis
VOCs	SW-846 8260C	Encore® /Terracore® Kits or lab supplied kits; bulk soil sample collected in 1- 4oz glass jar for dry weight determination	Cool to <6 °C; low-level freeze within 48 hours of collection; medium-level methanol	14 days from collection to analysis
SVOCs, including 1,4-Dioxane	SW-846 8270E/8270E-SIM	1- 8oz glass jar with Teflon®-lined lid	Cool to <6 °C	14 days from collection to extraction, 40 days from extraction to analysis
Metals	SW-846 6010D	1- 4oz plastic or glass bottle	Cool to <6°C	180 days from collection to analysis (inclusive of digestion and analysis)
Mercury	SW-846 7471B	1- 4oz plastic or glass bottle	Cool to <6°C	28 days from collection to analysis (inclusive of digestion and analysis)
Hexavalent Chromium	SW-846 7196A	1- 4oz plastic or glass bottle	Cool to <6°C	30 days from collection to extraction, 168 hours from extraction to analysis
PCBs	SW-846 8082A	1- 4oz glass jar with Teflon®-lined lid	Cool to <6°C	14 days from collection to extraction, 40 days from extraction to analysis
Pesticides	SW-846 8081B	1- 4oz glass jar with Teflon®-lined lid	Cool to <6°C	14 days from collection to extraction, 40 days from extraction to analysis
Herbicides	SW-846 8151A	1-4oz glass jar with Teflon®-lined lid	Cool to <6°C	14 days from collection to extraction, 40 days from extraction to analysis

Notes:
1. USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste SW-846 3rd ed. Update IV Washington, D.C 1996*
ASTM International. ASTM Method D8421-22. 2022
USEPA. Method 1633 EPA 821-R-24-001. 2024
2. The laboratory should be consulted prior to sample collection, as it may be possible to combine sample volume for multiple analyses in one sample container.
3. All holding times are calculated from date of collection. It is imperative that all samples are submitted to the laboratory with ample time for the analysis to be completed within the holding time. Missing a holding time is unacceptable and may result in unusable data if the holding time is missed.

Abbreviations:
°C = degrees Celsius
HDPE = high density polyethylene
mL = milliliter
L = liter
oz = ounce
PCBs = polychlorinated Biphenyls
PFAS = per- and polyfluoroalkyl substances
PP = polypropylene
VOCs = volatile organic compounds
SVOCs = semi-volatile organic compounds

Appendix A

Field Quality Procedures and Technical Guidance Instructions

QP 3.06 – Field Activities Documentation

Rev: 2

Rev Date: July 19, 2023

Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
	0	November 8, 2016	All	QP Issued	QMS
	1	November 30, 2021	All	Updated Template for QMS Relaunch October 12, 2021	Matt Spurlin Brian Webb David Gerber
	2	July 19, 2023	All	Annual review completed by SME. Rev number and date updated.	Brian Webb
			Page 6	Deleted “Collector, Survey123” replaced with "ESRI Field Maps, ESRI Survey123"	

Approval Signatures

Prepared by:	Matt Spurlin / Brian Webb	7/19/2023
	Names (Preparers)	Date
Quality Reviewer:		7/19/2023
	Matt Spurlin / Brian Webb (Quality Reviewers)	Date
QMS Approver:		7/19/2023
	David Gerber (QMS Approver)	Date

STATEMENT OF POLICY:

It is Arcadis Environment Business Line (ENV) policy that field activities must be documented to facilitate the interpretation of data; show compliance with project plans, work plans, and contract terms; and to serve as evidentiary records. Documentation reflecting activities performed must be legible, organized, and complete. Applicable regulatory and client requirements should be considered when documenting field activities. Project-specific requirements for documentation typically should be described in the Work Plan, Field Sampling Plan (FSP), and/or in the Quality Assurance Project Plan (QAPP).

1 Purpose

The purpose of this Quality Procedure (QP) is to provide a standard procedure for the documentation of fieldwork activities. This documentation pertains to site-related projects, but is not limited to the collection of samples, subsurface information, and oversight of construction activities. Field documentation must include, at a minimum, project title and number, date and times of activities, the identification of the employee performing the work, and the specifics of the work being performed.

2 Responsibilities

Certified Project Manager (CPM) – is responsible for the project-related administration of this QP.

Quality Consultant – is responsible for providing quality assurance and quality control guidance to the CPM in implementing this procedure. Note that for federal projects, there are specific requirements and qualifications for the QA Officer assigned to the project.

Project Team Members – who are assigned to document field activities, are responsible for compliance with this procedure.

Quality Reviewer – The Quality Reviewer is responsible for final review of this Quality Procedure (QP). Quality Reviewers may be a Quality Consultant, QMS Document Owner, Technical Solution Leader, Community of Practice Leader, or another qualified subject matter expert (SME).

3 Terms and Conditions

Field Sampling Plan (FSP) – A document that describes the procedures and protocols necessary to complete field sampling and data collection activities.

Work Plan – A document that describes proposed project activities.

Quality Assurance Project Plan (QAPP) – A document that prescribes the quality assurance/quality control (QA/QC) procedures to be followed. Uniform Federal Policy (UFP) QAPPs are now frequently required for environmental projects by most federal regulatory agencies. A UFP QAPP includes Worksheets used to document the entire project plan developed following the systematic planning process. For more details on the UFP QAPP see http://www.epa.gov/fedfac/documents/qualityassurance.htm. Note that if the project QAPP is written following the UFP format, it will also contain a description of the sampling rationale and sampling locations as well as QA/QC requirements. The UFP QAPP format is designed to capture the entire systematic planning

process. If a UFP QAPP is written for a project, a separate FSP is generally not required unless specified by the particular client or contract.

Technical Guidance Instruction (TGI) – Document describes the procedure and/or protocol necessary to conduct a specific activity.

4 Related Documents

Forms used for documenting field activities may be included as attachments to the FSP or the QAPP and may include the following examples:

- Chain-of-custody (COC) form;
- Sample data log;
- Field modification form;
- Sample receipt form;
- Corrective action form;
- Field activity log;
- Calibration log;
- Analysis request and chain-of-custody record;
- Daily quality control reports;
- Purge log;
- Soil boring log.

Examples of TGIs with forms and check-lists can be found in the QMS Document Library.

5 Description of the Procedure

1. General Requirements

1.1 Documentation Format

Documentation of field activities provides an accurate and comprehensive record of the work performed sufficient for a technical peer to reconstruct the day's activities and confirm that necessary client, regulatory, contract, and work plan requirements were met. General requirements include:

- Use of field books (preferably bound) as the primary source for information collection and recording. Field books should be dedicated to the project and appropriately labeled.
- Use of a Field Activity Log is suggested to formally document activities and events as a supplement to bound field books. The Field Activity Log can be a standard or project-specific form or a bound field book. Preprinted standard forms are available for many activities and should be used whenever possible. These forms will provide prompts and request additional information that may be useful and/or needed. Project-specific field forms may be generated, or existing forms may be modified to meet specific project needs. Client-supplied forms may be substituted, as required.
- Appropriate header information is documented on the first page of notes for each day of fieldwork, including project title, project number, date, time, author, and relevant setting information such as weather conditions, topography, surface water conditions, observed site activities/uses, and other persons in field team. In addition, include on every page of notes the page number and date. Project-specific information depends on

the nature of work being performed and should be discussed by the project team prior to commencing fieldwork. As appropriate, dedicated field logs/journals or forms should be used. When Field Activity Log Forms are used, information fields that are not applicable should be noted as such with the symbol "N/A" or other appropriate notation.

- Field documentation entries shall be made using indelible ink.
- Data entries shall be legible. A single line should be drawn through incorrect entries and the corrected entry written next to the original strikeout. Strikeouts are to be initialed and dated by the originator.
- Units of measurement shall be specified. The level of accuracy shall be indicated (e.g., observed estimate vs. quantified census from direct count).
- Field records are to be maintained in project files unless otherwise specified by a client or stipulated by a contract.
- Unless addressed specifically by a client or stipulated by a contract, site photographs should be taken to document the general setting and landscape as well as site-specific issues/resources of interest. Photo locations and the compass direction of view should be recorded in the notes with the photo number.
- Alternatively, use of field tablets with data plans and electronic data collection software supported by Cloud services (e.g., Fulcrum, ESRI Field Maps, ESRI Survey123) that are pre-loaded with appropriate data collection forms (e.g., method-specific sampling, soil logging, photo logs, H&S tailgate meetings, well/piezometer/lysimeter installations, electronic chain of custody, etc.). All forms adhere to the relevant documentation format requirements (described above and below this section) for data collection with conventional paper methods. Electronic data collection services, as well as training, are available to Arcadis field staff through the FieldNow® program and while optional are highly encouraged.

1.2 Documentation Entries

A chronology of field events should be recorded. General entry requirements include:

- Visitors to the site, including owner and regulatory agency representatives
- Summary of pertinent project communications with the client, regulators, or other site visitors during the fieldwork
- Other contractors or entities working on site
- A description of the day's field activities, generally in chronological sequence or in order of significance, using military time notation (e.g., 9:00 a.m. as 0900, and 5:00 p.m. as 1700)
- If applicable, calibration of measuring and test equipment and identification of the calibration standard(s) (use a Calibration Log, if available, with cross-reference entered into the field book)
- Field equipment identification, including information such as the type, manufacturer, model number, or other specific information
- If applicable sampling activities are being performed, weather information such as temperature, wind speed and direction, precipitation, time of measurement, and units
- Documentation of safety meeting (e.g., tailgates and tailboards) topics and attendees
- Verification of subsurface utility clearance in accordance with ENV policy
- Safety and/or monitoring equipment readings, including time of measurements and units
- If applicable, specific forms used for collection of data are referenced in the field notebook
- Subcontractor progress and/or problems encountered
- Changes in the scope of work
- Other unusual events.

2. Specific Requirements

2.1 Sample Collection

Sample collection data are documented in a bound field book, electronic field forms provided by the FieldNow® program, and/or on a Field Activity Log. Where both are being used, information contained in one is cross-referenced to the other. Entries such as the following examples should be consistent with the requirements in the project-specific Work Plan, FSP, and QAPP:

- Sample identification number, location taken, depth interval, sample media, sample preservative, collection time, and date
- Sample collection method and protocol
- Physical description of the sample (using a standard classification system for soil)
- If a composite sample, include the number, location(s), and depth(s) of grab samples incorporated in the composite
- Quality-related samples (e.g., field duplicates, trip blanks, equipment rinse, blanks matrix spikes, and matrix spike duplicates)
- Container description and sample volume
- Pertinent technical data, such as pH, conductivity, temperature, and head-space readings
- Pertinent technical comments
- Identification of personnel collecting the sample.

2.2 Sample Labeling

Sample labels must be prepared and attached to sample containers. Labels are either provided by the laboratory performing the analyses or are generated internally. Labels should be indelible and securely attached to the container. The information to be provided may include:

- Sample identification number
- Sample date, initials, or name of who collected the sample, and collection time
- Physical description of the sample (e.g., water, solid, gas, or other physical medium)
- Analytical parameters and method(s)
- Preservatives, if present
- Sample location and depth, if applicable
- Client.

Although this information is typically written out, it can also be recorded in an electronic tracking system if a bar code is used.

2.3 Analysis Request and Chain-of-Custody Record

A critical component of data collection is the documentation that the samples were obtained from specific locations and received by the laboratory or archive without alteration. Evidence of collection, shipment, laboratory receipt, and laboratory custody until disposal or archive must be properly documented. Documentation will be accomplished through a COC record that documents each sample and identifies the individuals responsible for sample collection, shipment, and receipt. A sample is considered in custody if at least one of the following criteria is met:

- The sample is in a person's actual possession
- The sample is in unobstructed view, after being in the person's actual possession
- The sample is locked and only accessible by the custodian after having been in the person's actual possession
- The sample is in a secured area, restricted to authorized personnel (e.g., laboratory).

An example COC form to be used by ENV personnel in collecting and shipping samples can be found on the corporate Intranet. A laboratory typically will not accept samples for analysis without a correctly prepared COC form. The COC must be signed by each individual who has the sample in his/her custody. Each sample shipped to a laboratory for analyses must be documented on the COC. Information on this form correlates with other supporting documentation, including the field logbook, sample labels, and sample collection logs.

The COC documents the elapsed time and the custodians of the sample from the time of its collection. The individuals who have physically handled the sample(s) or witnessed initial sample collection and packaging (sample team member) must be identified on the form. A sample team member relinquishes the sample by signing the COC. Individuals who either relinquish or receive samples must include their complete names, company affiliation, and the date and time the sample(s) were relinquished. The times that the samples are relinquished and received by the next custodian should coincide, with the exception of transfer by commercial carriers. These carriers will not be required to sign the COC.

If a sample is to be stored for a period of time (e.g., overnight), measures are taken to secure the sample container in a manner that only provides access to the custodian of record. If samples are relinquished to a commercial carrier (i.e., UPS or Federal Express), the carrier waybill number is recorded, and a copy of the waybill is attached to the COC. These documents are maintained with other field documentation. The original COC is sealed inside a zip-top plastic bag and placed inside the shipping container with the samples.

If corrections are made to the COC, the corrections should be made (single line through the error, initial, and date) by the originator of the change, and, if necessary, an explanation of the change should be provided. The documentation should be of a level of detail that clearly documents the change to a third-party reviewer.

Guidance for choosing a laboratory and completing analyses requests and COC can be found in QP 2.09-Subcontracting Laboratory Services and on the corporate Intranet for the Arcadis Laboratory Program (ALP) and should also be described in the project-specific planning documents (i.e., Work Plan, FSP. or QAPP).

The option to use the Electronic Chain of Custody (eCOC) form in conjunction with the appropriate sample application(s) may be available through the FieldNow® program but is currently limited to a select list of approved analytical laboratories. Use of the eCOC application is intended to reduce common transcription errors both by field staff and laboratory staff on a conventional handwritten paper COC. Once the eCOC form is completed and approved on the field tablet by field staff, a PDF version of the form is automatically emailed to each assigned team member. In addition, a dedicated or mobile printer is recommended for printing a hard copy of the completed eCOC to be included in each sample cooler to meet laboratory requirements.

2.4 Subsurface Logs

Test pits, soil borings, monitoring wells or rock coreholes wells, and piezometer installations are to be recorded in bound field books or electronic soil logging field form provided by the FieldNow® program and may be supplemented with prepared forms. Personnel completing the log are to supply the following information:

- Administrative and technical information included in the header.
- Types of equipment used (e.g., drill rig type, drilling tools used [including diameter and length], or backhoe model).
- Subcontractor/driller used.
- Descriptions of subsurface materials encountered, and the number and type of samples collected, if any.
- Subsurface exploration depth and units of measure.
- For drilling, length of recovery.
- Sample type and sample number for geotechnical or analytical samples collected. These data are to be also entered on the sample collection log (if used) and the sample label.
- Classification standard protocol used, if any (e.g., ASTM International Standard Penetration Test).
- Narrative description of the soil, sediment, or bedrock (using standard classification system) and other pertinent information.
- Additional data, such as background and sample vapor/gas readings, observation of sheens, non-aqueous-phase liquid, depth to water (if encountered), presence of (but generally not description of) odors, changes in drilling conditions, and other pertinent information.
- Description of the materials used to seal the boring unless it is completed as a well or piezometer.

When using the electronic soil logging field form, draft logs can be exported to appropriate formats through Arcadis's internal reporting services for QC. Revisions can be made to the soil logging forms using the field tablets, then re-synced to the server and re-exported to PDF as final draft.

2.5 Monitoring Well/Piezometer Installation

In addition to requirements in Section 2.4, subsequent well or piezometer development activities may involve transcription of field data from the field book onto a computer-based boring log. The field notebook or electronic soil logging/well construction field form provided by the FieldNow® program is to be used to identify the chronology and major events of the installation activity, and the computer-based boring log is to be used to correlate the geologic strata to the major elements of the monitoring well construction. Information to be collected and recorded must meet the regulatory and client requirements and may include the following:

- Location identity
- Screen and riser type, length, diameter, and location
- Diameter
- Total depth
- Sump location and depth and diameter
- Materials of construction (e.g., stainless steel, polyvinyl chloride, or other material)
- Seal type(s) and or depth(s)
- Sand or gravel pack type, including materials (e.g., silica) and gradation
- Depth to water before and after installation.

When using the electronic soil logging/well construction field form, draft logs can be exported to appropriate formats through Arcadis's internal reporting services for QC. Revisions can be made to the soil logging/well construction forms using the field tablets, then re-synced to the server and re-exported to PDF as final draft.

2.6 Air Sampling Logs

At a minimum, air sampling documentation should include:

- Start and finish time of sampling
- Sampling location
- Sampling method/media
- Volume sampled.

2.7 Construction, Demolition, Abandonment, and Related Activities

Monitoring and documentation of construction and comparable activities shall be documented in bound field books and/or on appropriate company forms and should include similar information as specified above, including information such as:

- Project name and number
- Owner or client name
- Contractor or subcontractors performing the work
- Contractor or subcontractor superintendent(s) and personnel (as available) on site
- Chronological sequence and description of work activities performed, including workday start and completion times
- Reference to contract sections, work plans, or specifications describing work being performed
- Reference to relevant permit conditions and regulatory requirements and/or reference to regulatory guidance documents controlling work approach
- Listing of all trades performing work by contractor and subcontractor
- Hours worked per trade
- Work hours per day per shift, if applicable
- Equipment on site (e.g., description, model number, size, and type) and hours of use
- Listing of equipment on site being left idle
- Description and quantity of materials used or incorporated, with reference to contract or specification item number, if feasible; include simple sketch of excavation with approximate dimension, if applicable
- Calculations with dimensions for quantities of material used or incorporated
- Delineation of the work area and access routes (e.g., fencing, flagging, or staking), confirmation that activities occurred within the work area or description of work occurring outside the delineated work area and justification (as needed), and characterization of impacts outside the designated work area
- Documentation of compliance with speed limits, dust control, erosion control best management practices, and other basic elements of construction activities as dictated by project work plans and applicable permits and regulatory criteria.

2.8 Daily Safety Meeting

A Daily Safety Meeting is to be conducted and documented each workday prior to the initiation of field activities, with on-site ENV personnel, contractors, subcontractors, and visitors if possible. Safety topics discussed are entered on the Daily Safety Meeting Form (available on the corporate Intranet). Topics discussed should include site-specific conditions, procedures to be followed that day, and protective equipment. A printed listing of the attendees at the meeting and their signatures should be included. Other required data are:

- Identification of the individual conducting the meeting and his/her signature
- Identification of the project supervisor and project manager.

The option to use the electronic H&S tailgate meeting field form is available through the FieldNow® program. The completed form can be exported to appropriate formats through Arcadis's internal reporting services for documentation purposes.

2.9 Calibration

Documentation of the calibration and calibration results shall be made for field equipment requiring calibration measuring and test equipment calibration data are recorded in the field book or on the Field Activity Log. Calibration data include the following:

- Unique identification of instrument being calibrated, including type, model, and serial number
- Date and time of calibration
- Standards used in the calibration, including standard identity, concentration, lot number, and manufacturer of the standard
- Instrument reading with respect to each calibration standard
- Comments, as necessary, regarding instrument performance.

2.10 Photographs and Videos

When the client allows, photographs and videos may be used to help document pre-, active, and post-field activities. In sensitive areas (e.g., secured, or confidential), the client must be contacted to evaluate security procedures concerning use of photographs or videos. Photographic and video documentation should include project title, project number, date, time, and description of conditions. The time should also be documented if time is important to a sequence of photographs.

Photographs are documented by numbering digital photographs and identifying the number and subject on the Field Activity Log or the electronic photograph log through the FieldNow® program. Individual prints may be marked with a stamp or preprinted self-adhesive labels, or by writing the project number and sequential number of each photograph and referencing the numbers in the field book, the Field Activity Log, or a dedicated photo log. Videos used for field documentation are to be identified by project title, project number, and description.

2.11 Subcontractor Preparedness Checklist

Prior to starting work, a review is to be made and documented of a subcontractor's preparedness to perform specified activities. This review may be documented on the Field Activities Log or on checklists that may be developed according to requirements for subcontracted work activities. Particular emphasis should be on site-specific issues that may require special consideration such as health and safety, access, and unique settings. These should be discussed in advance with the CPM and the client in developing and implementing the Scope of Work.

- END OF PROCEDURE -

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TGI – Groundwater and Soil Sampling Equipment Decontamination

Rev: 3

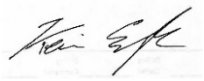
Rev Date: August 30, 2023

Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
	0	February 23, 2017	All	Conversion from SOP to TGI	Cassandra McCloud / Pete Frederick
	1	May 8, 2020	4, 5	Added note regarding use of Liquinox and 1,4-Dioxane	Marc Killingstad
	2	June 14, 2022	All	Conversion to new TGI format and minor edits.	Kevin Engle / Marc Killingstad
	3	August 30, 2023	All	Annual review completed by SME. Updated Rev 1.	Marc Killingstad

Approval Signatures

Prepared by: 8/30/2023



Name (Preparer) Date

Reviewed by: 8/30/2023



Marc Killingstad (Subject Matter Expert) Date

1 Introduction

This document is intended to provide guidance to staff performing decontamination procedures at project sites. The content in this document describes the intended use, scope and application, personnel qualifications, equipment, cautions, health and safety considerations, procedures, waste management, data recording and management, and quality assurance of decontamination procedures.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

Decontamination is performed on sampling equipment prior to sample collection to ensure that the sampling equipment that contacts a sample, or monitoring equipment that is brought into contact with environmental media to be sampled, is free from analytes of interest and/or constituents that could interfere with laboratory analysis for analytes of interest. Sampling equipment must be appropriately cleaned prior to use for sampling or coming into contact with environmental media to be sampled and following completion of the sampling event prior to shipment or storage. The effectiveness of the decontamination procedure should be verified by collecting and analyzing equipment blank samples.

The sampling equipment cleaning procedures described herein includes pre-field, in the field, and post- field cleaning of sampling equipment which may be conducted at an established equipment decontamination area (EDA) on site, as appropriate and necessary. Sampling equipment that may require decontamination at a given site include soil sampling tools; groundwater, sediment, and surface-water sampling devices; water testing instruments; down-hole instruments; and other activity-specific sampling equipment. Non-disposable equipment will be cleaned before collecting each sample, between each sample collected, and prior to placing sampling equipment in protective cases, or containers for transport. Cleaning procedures for sampling equipment should be monitored by collecting equipment blank samples as required in project work plans, field sampling plans, quality assurance project plans (QAPP), or other pertinent project documents. Dedicated and/or single-use (i.e., not to be re-used) sampling equipment will not require decontamination.

4 Personnel Qualifications

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour hazardous waste operations and emergency response (HAZWOPER) training and/or Occupational Safety and Health Administration (OSHA) HAZWOPER site supervisor training. Arcadis personnel will also have current training as specified in the Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. In addition, Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and Technical Guidance Instructions (TGIs) and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The project HASP and other documents will identify other training requirements or access control requirements.

5 Equipment List

The equipment required for equipment decontamination is presented below. Note that certain contaminants may require specific materials be used that are not captured in this list. Always review project and contaminant specific TGIs or work plans to ensure proper equipment is utilized. Note for per- and polyfluoroalkyl substances (PFAS) see *TGI – Per- and Polyfluoroalkyl Substances (PFAS) Field Sampling Guide*.

- Health and safety equipment, including appropriate personal protective equipment (PPE), as required in the site HASP
- Deionized water that meets the analytical criteria for deionized water with no detectable constituents above the reporting limits for the methods to be used and analytes being analyzed for. Deionized water is used for inorganics, and organic-free water for volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), pesticides, etc.
- Non-phosphate detergent such as Alconox® or, if sampling for phosphorus or phosphorus- containing compounds, Liquinox (or equivalent). NOTE: Liquinox has shown to provide false positives for 1,4-Dioxane and should not be used at sites where that may be a constituent of concern (COC).
- Tap water
- Rinsate collection plastic containers

- Department of Transportation (DOT)-approved waste shipping container(s), as specified in the work plan, field sampling plan, or regulatory requirements if decontamination waste is to be shipped for disposal
- Brushes
- Large heavy-duty garbage bags
- Spray bottles
- (Optional) – Isopropyl alcohol (free of ketones) or methanol. These can be wipes or diluted with water (usually 1part isopropyl/methanol to 10 parts water) if a spray is needed.
- Airtight, sealable plastic baggies, such as Ziploc®-type
- Plastic sheeting

6 Cautions

Rinse equipment thoroughly and allow the equipment to dry before re-use or storage to prevent introducing solvent into sample medium. If manual drying of equipment is required, use clean lint-free material to wipe the equipment dry. Ensure all rinse materials do not adversely affect sample collection efficiency or analytical results.

Store decontaminated equipment in a clean, dry environment. Do not store near combustion engine exhausts. Properly containerize equipment to ensure cross-contamination doesn't happen from other uncontaminated surfaces or equipment.

If equipment is damaged to the extent that decontamination is uncertain due to cracks, gouges, crevices, or dents, the equipment should not be used and should be discarded or submitted for repair prior to use for sample collection.

A proper shipping determination regarding hazardous materials will be performed by a DOT-trained individual for cleaning materials shipped by Arcadis.

Caution should be exercised to avoid contact with the pump casing and water in the container while the pump is running (do not use metal drums or garbage cans) to avoid electric shock.

7 Health and Safety Considerations

Review the safety data sheets (SDS) for the cleaning agents and materials used in decontamination. If solvent is used during decontamination, use appropriate PPE and work in a well-ventilated area and stand upwind while applying solvent to equipment. Apply solvent in a manner that minimizes potential for exposure to workers and bystanders. Follow health and safety procedures outlined in the HASP.

8 Procedure

A designated area will be established to clean sampling equipment in the field prior to and following sample collection. Equipment cleaning areas will be set up within or adjacent to the specific work area, but not at a location that expose equipment to contamination (i.e., exposed to combustion engine exhaust). Detergent solutions will be prepared in clean containers for use in equipment decontamination. Decontaminated equipment

will be handled by workers wearing clean gloves, properly changed to prevent cross-contamination. The procedures detailed in this section provide an overview of common decontamination techniques. Additional steps may be required based on the type of contaminant present or client/site requirements.

Cleaning Sampling Equipment

1. Wash the equipment/pump with potable water.
2. Wash with detergent solution (Alconox®, Liquinox® or equivalent) to remove all visible particulate matter and any residual oils or grease. NOTE: Liquinox® has shown to provide false positives for 1,4-Dioxane and will not be used at sites where that may be a COC.
3. If equipment is very dirty, precleaning gross debris with a brush and tap water may be necessary.
4. If non-aqueous phase liquids are present, the use of isopropyl alcohol (free of ketones) or methanol is recommended. Cloth wipes or diluted solution can be used to remove the non-aqueous phase liquids that are hard to remove with detergent solution in step 2. Consult with project manager if non-aqueous phase liquids are present onsite and design an appropriate decontamination procedure that includes step 4.
5. Rinse with deionized water.

Decontaminating Submersible Pumps

Submersible pumps may be used during well development, groundwater sampling, or other investigative activities. The pumps must be cleaned and flushed before and between uses. This cleaning process will consist of an external detergent solution wash and tap water rinse, a flush of detergent solution through the pump, followed by a flush of potable water through the pump. Flushing will be accomplished by using an appropriate container filled with detergent solution and another container filled with potable water. The pump will be be flushed with deionized water as the last step prior to use. The pump will run long enough to effectively flush the pump housing and hose (unless new, disposable hose is used). Disconnect the pump from the power source before handling. The pump and hose will be placed on or in clean polyethylene sheeting to avoid contact with the ground surface.

9 Waste Management

Equipment decontamination rinsate will be managed in conjunction with all other waste produced during the field sampling effort. Waste management procedures are outlined in the work plan or Waste Management Plan (WMP).

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff

are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Equipment cleaning and decontamination will be noted during project documentation. Information will include the type of equipment cleaned, the decontamination location, specific procedures utilized, solvents and/or cleaning agents used, source of water, and deviations or omissions from this TGI.

Unusual field conditions should be noted if there is potential to impact the efficacy of the decontamination or subsequent sample collection.

An inventory of the solvents brought on site and used and removed from the site will be maintained in the project documentation. Records will be maintained for solvents used in decontamination, including lot number and expiration date.

Containers with decontamination fluids will be labeled.

11 Quality Assurance

Equipment blanks should be collected to verify that the decontamination procedures are effective in minimizing potential for cross contamination. The equipment blank is prepared by pouring deionized water (or organic-free water, for organic analyses) over the clean and dry tools and collecting the water into appropriate sample containers. Equipment blanks should be analyzed for the same set of parameters that are performed on the field samples collected with the equipment that was cleaned as specified in the sampling and analysis plan. Equipment blanks are collected per equipment set, which represents all the tools needed to collect a specific sample.

12 References

USEPA Region 9 - Field Sampling Guidance #1230, Sampling Equipment Decontamination.

USEPA Region 1 - Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells.

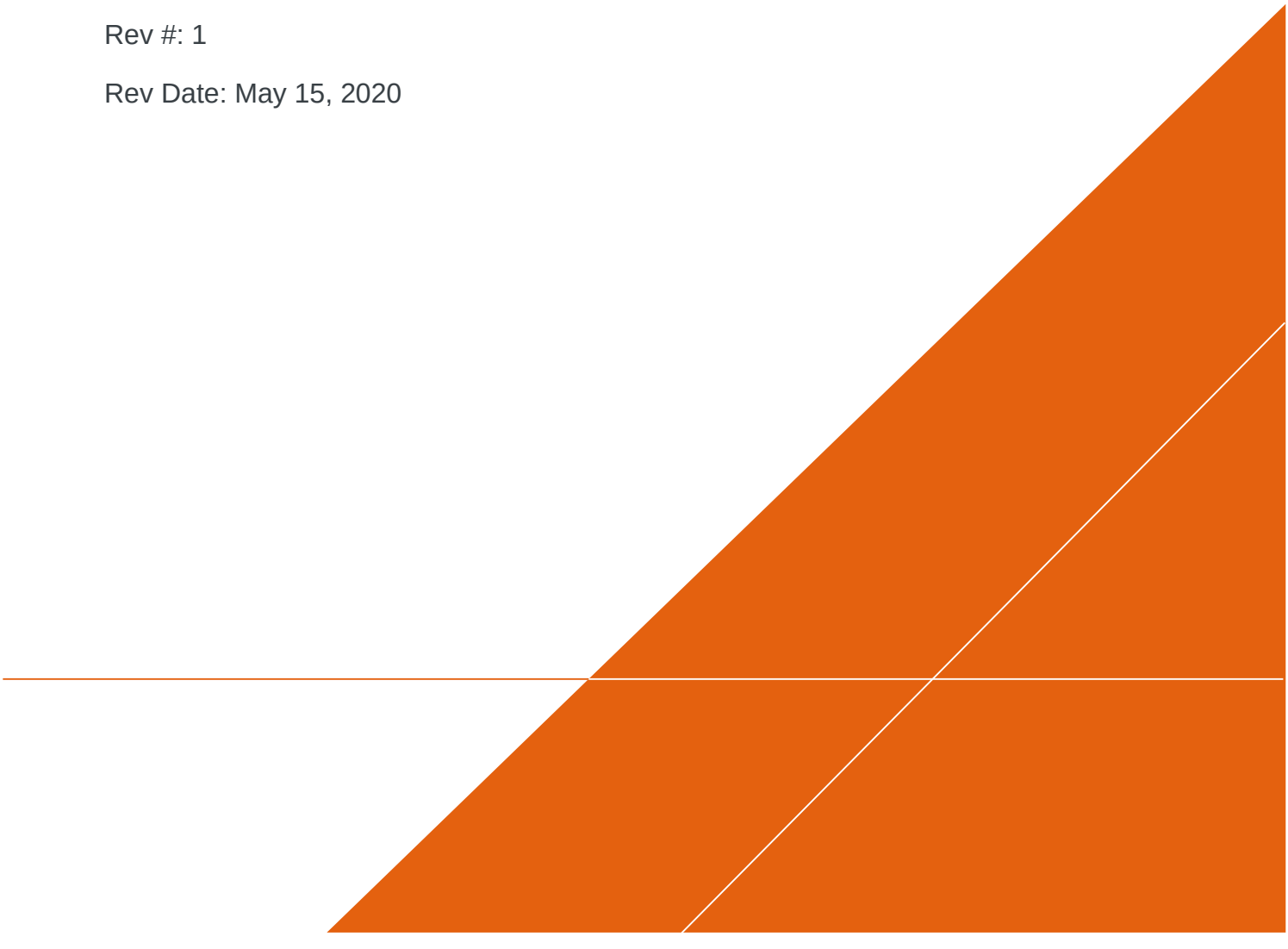
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ARCADIS Design & Consultancy for natural and built assets

TGI - INVESTIGATION-DERIVED WASTE HANDLING AND STORAGE

Rev #: 1

Rev Date: May 15, 2020



VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	February 23, 2017	ALL	Conversion from SOP to TGI	Ryan Mattson / Peter Frederick
1	May 15, 2020	ALL	Updated to reflect regulatory changes	

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APPROVAL SIGNATURES

Prepared by:		02/23/2017
	Derrick Maurer	Date:
Technical Expert Reviewed by:		05/15/2020
	Ryan Mattson (Technical Expert)	Date:

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1 INTRODUCTION

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2 SCOPE AND APPLICATION

The objective of this Technical Guidance Instruction (TGI) is to describe the procedures to manage investigation-derived wastes (IDW), both hazardous and nonhazardous, generated during site activities, which may include, but are not limited to: drilling, trenching/excavation, construction, demolition, monitoring well sampling, soil sampling, decontamination and remediation. For the purposes of this TGI, IDW is considered to be discarded materials which are defined as solid waste by United States Environmental Protection Agency (EPA) standard 40 CFR § 261.2 (which may include liquids, solids, or sludges). IDW may include soil, groundwater, drilling fluids, decontamination liquids, as well as contaminated personal protective equipment (PPE), sorbent materials, construction and demolition debris, and disposable sampling materials. Hazardous or uncharacterized IDW will be collected and staged at the point of generation. Quantities small enough to be containerized in 55-gallon drums will be taken to a designated temporary onsite storage area (discussed in further detail under Drum Storage) pending characterization and disposal. IDW materials will be characterized using process knowledge and appropriate laboratory analyses to determine the waste classification and evaluate proper safe handling and disposal methods.

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This TGI describes the necessary equipment, field procedures, materials, regulatory references, and documentation procedures necessary for proper handling and storage of IDW up to the time it is properly transported from the project site and disposed. The procedures included in this TGI for handling and temporary storage of IDW are based on the EPA's guidance document *Guide to Management of Investigation Derived Wastes* (USEPA, 1992). IDW is assumed to be contaminated with the site constituents of concern (COCs) until analytical evidence indicates otherwise. IDW will be managed to ensure the protection of human health and the environment and will comply with all applicable or relevant and appropriate requirements (ARAR). Although not comprehensive, the following laws and regulations on Hazardous Waste Management should be considered as potential ARAR. It is the Arcadis Certified Project Manager (CPM) and/or designated Technical Expert to determine which laws and regulations, at all levels of government, are applicable to each project site and activity falling under this TGI.

Federal Laws and Regulations

- Resource Conservation and Recovery Act (RCRA) 42 USC § 6901-6987.
- Federal Hazardous Waste Regulations 40 CFR § 260-265

Department of Transportation (DOT) Hazardous Materials Transportation 49 CFR

Occupational Safety and Health Administration (OSHA) Regulations 29 CFR

State Laws and Regulations

- To be determined based on location of site and location of treatment, storage, and/or disposal facility (TSDF) to be utilized.

Regional, County, Municipal, and Local Regulations

- To be determined based on location of site and location of treatment, storage, and/or disposal facility (TSDF) to be utilized.

Initial Storage

Pending characterization, IDW will be temporarily stored appropriately within each area of contamination (AOC). Under RCRA, "storage" is defined as the "holding of hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere" (40 CFR § 260.10). The onsite waste staging area will be in a secure and controlled area. Uncharacterized wastes are considered potentially hazardous wastes and must be stored in DOT approved packaging. Liquid wastes must be stored in DOT approved closed head drums or other approved containers (e.g., portable tank containers) that are compatible with the type of material stored therein. Solid materials must be stored in DOT approved open head drums where practicable. Larger quantities of solid IDW can be containerized in bulk containers (such as in a roll-off box). Soil from large excavation projects may be managed in stockpiles with within the AOC and does not need to be containerized until exiting the AOC.

Characterization

Waste characterization can either be based on generator knowledge, such as using historical process knowledge and safety data sheets (SDS), or can be based upon characterization sampling analytical results. IDW typically is not characterized using SDS as it is a mixture of aged chemicals and environmental media. Historical process knowledge should be used to determine if the IDW is a listed hazardous waste (40 CFR § 261.31-33). If the IDW is not a listed hazardous waste, waste

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characterization can be completed by laboratory analysis of representative samples of the IDW. The laboratory used for waste characterization analysis must have the appropriate state and federal accreditations and may be required to be pre-approved by the Client. IDW will be classified as RCRA hazardous or non-regulated under RCRA based on the waste characterization determination.

If IDW is characterized as RCRA hazardous waste, RCRA and DOT requirements must be followed for packaging, labeling, transporting, storing, and record keeping as described in 40 CFR § 262 and 49 CFR § 171-178. Waste material classified as RCRA nonhazardous may be handled and disposed of as nonhazardous waste in accordance with applicable federal, state, and local regulations.

Storage Time Limitations

Containerized hazardous wastes can be temporarily stored for a maximum of 90 calendar days from the accumulation start date for a large quantity generator or a maximum of 180 calendar days from the accumulation start date for a small quantity generator. Wastes classified as nonhazardous may be handled and disposed of as nonhazardous waste and are not subject to storage time limitations.

This is TGI may be modified by the CPM and/or Technical Expert for a specific project or client program, as required, dependent upon client requirements, site conditions, equipment limitations, or limitations imposed by the procedure. The resulting procedure employed to execute the work will be documented in the project work plans or reports. If changes to the sampling procedures are required due to unanticipated field conditions, the changes will be discussed with the CPM and/or Technical Expert as soon as practicable, and if approved to be performed, be documented.

3 PERSONNEL QUALIFICATIONS

Arcadis field sampling personnel will have current regulatory- and Arcadis-required health and safety training including 40-hour HAZWOPER training, site supervisor training, site-specific training, first aid, and cardiopulmonary resuscitation (CPR), as needed. Personnel handling and packaging hazardous waste and performing hazardous waste characterizations must have RCRA hazardous waste management training per 40 CFR § 264.16. Additional state-specific hazardous waste management training is required in certain states (i.e., California).

Although not common practice, in certain situations Arcadis personnel may sign waste profiles and/or waste manifests on a case by case basis for clients, provided the appropriate agreement is in place between Arcadis and the client documenting that Arcadis is not the generator, but is acting as an authorized representative of the generator. Arcadis personnel who sign waste profiles and/or waste manifests will have both current RCRA hazardous waste management training per 40 CFR § 264.16 and current DOT hazardous materials transportation training per 49 CFR § 172.704. Arcadis field personnel will also comply with client-specific training. In addition, Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and Technical Guidance Instructions (TGIs) and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The project health and safety plan (HASP) and other documents will identify other training requirements or access control requirements.

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4 EQUIPMENT LIST

The Following Materials, as required, will be available for IDW handling and Storage:

- Appropriate personal protective equipment as specified in the Site Health and Safety Plan (HASP)
- DOT approved containers
- Hammer
- Leather gloves
- Drum dolly
- Appropriate drum labels (outdoor waterproof self-adhesive)
- Portable tank container
- Appropriate labeling, packing, chain-of-custody forms, and shipping materials as determined by the CPM and/or Technical Expert.
- Indelible ink and/or permanent marking pens
- Plastic sheeting
- Appropriate sample containers, labels, and forms
- Stainless-steel bucket auger
- Stainless steel spatula or knife
- Stainless steel hand spade
- Stainless steel scoop
- Digital camera
- Field logbook

5 CAUTIONS

Filled drums can be very heavy, become unbalanced, or spill its contents. Therefore, use appropriate moving techniques and equipment for safe handling. Similar media (e.g. soils with other soils; or liquids with other liquids) will be stored in the same drums to aid in sample analysis and disposal. Drum lids must be secured to prevent rainwater from entering the drums and leakage during movement. Drums containing solid material may not contain any free liquids. Waste containers stored for extended periods of time may be subject to deterioration. Drum Over Packs may be used as secondary containment. All drums must be visually inspected for condition to ensure that they are in good condition without visible evidence of rusting, holes, breakage, etc., to prevent potential leakage and facilitate subsequent disposal. All drum lids must be verified as having a properly functioning secured lid prior to use.

6 HEALTH AND SAFETY CONSIDERATIONS

As determined by the site's known and suspected hazards, appropriate PPE must be worn by all field personnel within the designated work area. Exposure air monitoring may be required during certain field activities as required in the Site Health and Safety Plan. If soil excavation in areas with potentially hazardous contaminants is possible, contingency plans will be developed to address the potential for encountering gross contamination or non-aqueous phase liquids. All excavation activities shall be in compliance with OSHA standard 29 CFR 1926.651 Excavations, and any other applicable regulations.

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Arcadis field personnel and subcontractors will be trained in and perform their work in compliance with all applicable federal, state, and local health and safety regulations as well as Arcadis' HASP and applicable Client health and safety requirements.

7 PROCEDURE

Specific waste temporary storage and handling procedures to be used are dependent upon the type of generated waste, including type of media (e.g. soils or free liquids) and constituents of concern. For this reason, IDW can be stored in a secure location onsite in separate 55-gallon storage drums, where solids can be stockpiled onsite (if nonhazardous) and purge water may be stored in portable tank containers. Waste materials such as broken sample bottles or equipment containers and wrappings will be stored in 55-gallon drums unless they were not in contact with sample media.

Management of IDW

Minimization of IDW should be considered by the project team during all phases of the project. Site managers may want to consider techniques such as replacing solvent based cleaners with aqueous-based cleaners for decontamination of equipment, reuse of equipment (where it can be properly decontaminated), limitation of traffic between exclusion and support zones, and drilling methods and sampling techniques that minimize the generation of waste. Alternative drilling and subsurface sampling methods may include the use of small diameter boreholes, as well as borehole testing methods such as a core penetrometer or direct push technique instead of coring.

Drum Storage

Drums containing hazardous waste will be stored in accordance with the requirements of 40 CFR 265 Subpart I (for containers) and 265 Subpart DD (for containment buildings). All 55-gallon drums will be stored at a secure, centralized onsite location that is readily accessible for vehicular pick-up. Drums confirmed as, or assumed to contain hazardous waste will be stored over an impervious surface provided with secondary spill containment. The storage location will, for drums containing liquid, have a containment system that can contain at least the larger of 10% of the aggregate volume of staged materials or 100% of the volume of the largest container. Drums will be closed during storage and be in good condition in accordance with the Guide to Management of Investigation-Derived Wastes (USEPA, 1992).

Hazardous Waste Determination

Waste material must be characterized to determine if it meets any of the federal definitions of hazardous waste as required by 40 CFR § 262.11. If the waste does not meet any of the federal definitions, it must then be established if any state-specific or local-specific hazardous waste criteria exist/apply.

Generator Status

Once hazardous waste determination has been made, the generator status will be determined. Large quantity generators (LQG) are generators who generate more than 1,000 kilograms of hazardous waste in a calendar month. Small quantity generators (SQG) of hazardous waste are generators who generate greater than 100 kilograms but less than 1,000 kilograms of hazardous waste in a calendar month. Very small quantity generators (VSQG) are generators who generate less than 100 kilograms of hazardous

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waste per month. Please note that a generator status may change from month to month and that a notice of this change is usually required by the generator's state agency.

Accumulation Time for Hazardous Waste

A LQG may accumulate hazardous waste on site for 90 calendar days or less without a permit and without having interim status, provided that such accumulation is in compliance with requirements in 40 CFR § 262.17. A SQG may accumulate hazardous waste on site for 180 calendar days or less without a permit or without having interim status, subject to the requirements of 40 CFR § 262.16. VSQG requirements are found in 40 CFR § 262.14. NOTE: The federal VSQG and SQG provisions may not be recognized by some states (e.g., California and Rhode Island). State-specific and local-specific regulations must be reviewed and understood prior to the generation of hazardous waste.

Satellite Accumulation of Hazardous Waste Satellite accumulation (SAA) will mean the accumulation of as much as fifty-five (55) gallons of hazardous waste, or the accumulation of as much as one quart of acutely hazardous waste, in containers at or near any point of generation where the waste initially accumulates, which is under the control of the operator of the process generating the waste, without a permit or interim status and without complying with the requirements of 40 CFR § 262.15 and without any storage time limit, provided that the generator complies with 40 CFR § 262.15.

Once more than 55 gallons of hazardous waste accumulates in SAA, the generator has three days to move this waste into storage.

Storage recommendations for hazardous waste include:

- Ignitable or reactive hazardous wastes must be >50 feet from the property line per 40 CFR § 265.176 (LQG generators only).
- Hazardous waste should be stored on a concrete slab (asphalt is acceptable if there are no free liquids in the waste).
- Drainage must be directed away from the accumulation area.
- Area must be properly vented.
- Area must be secure.

Drum/Container Labeling

Drums will be labeled on both the side and lid of the drum using a permanent marking pen. Old drum labels must be removed to the extent possible, descriptions crossed out should any information remain, and new labels affixed on top of the old labels. Other containers used to store various types of waste (e.g., polyethylene tanks, roll-off boxes, end-dump trailers, etc.) will be labeled with an appropriate "Waste Container" or "Testing in Progress" label pending characterization. Drums and containers will be labeled as follows:

- Appropriate waste characterization label (Pending Analysis, Hazardous, or Nonhazardous)
- Waste generator's name (e.g., client name)
- Project Name
- Name and telephone number of Arcadis project manager
- Composition of contents (e.g., used oil, acetone 40%, toluene 60%)
- Media (e.g., solid, liquid)
- Accumulation start date

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- Drum number of total drums as reconciled with the Drum Inventory maintained in the field log book.

IDW containers will remain closed except when adding or removing waste. Immediately upon beginning to place waste into the drum/container, a “Waste Container” or “Pending Analysis” label will be filled out to include the information specified above, and affixed to the container. Once the contents of the container are identified as either non-hazardous or hazardous, the following additional labels will be applied.

- Containers with waste determined to be non-hazardous will be labeled with a green and white "Nonhazardous Waste" label over the "Waste Container" label.
- Containers with waste determined to be hazardous will be stored in an onsite storage area and will be labeled with the "Hazardous Waste" label and affixed over the "Waste Container" label.

The ACCUMULATION DATE for the hazardous waste is the date the waste is first placed in the container and is the same date as the date on the "Waste Container" label. DOT hazardous class labels must be applied to all hazardous waste containers for shipment offsite to an approved disposal or recycling facility. In addition, a DOT proper shipping name will be included on the hazardous waste label. The transporter should be equipped with the appropriate DOT placards. However, placarding or offering placards to the initial transporter is the responsibility of the generator per 40 CFR § 262.33.

Inspections and Documentation

All IDW will be documented as generated on a Drum Inventory Log maintained in the field log book. The Drum Inventory will record the generation date, type, quantity, matrix and origin (e.g., Boring-1, Test Pit 3, etc.) of materials in every drum, as well as a unique identification number for each drum. The drum inventory will be used during drum pickup to assist with labeling of drums. The drum storage area and any other areas of temporarily staged waste, such as soil/debris piles, will be inspected weekly. The weekly inspections will be recorded in the field notebook or on a Weekly Inspection Log. Digital photographs will be taken upon the initial generation and drumming/staging of waste, and final labeling after characterization to document compliance with labeling and storage protocols, and condition of the container. Evidence of damage, tampering or other discrepancy should be documented photographically.

Emergency Response and Notifications

Specific procedures for responding to site emergencies will be detailed in the HASP. If the generator is designated as a LQG, a Contingency Plan will need to be prepared to include emergency response and notification procedures per 40 CFR § 265 Subpart D. In the event of a fire, explosion, or other release which could threaten human health outside of the site or when Client or Arcadis has knowledge of a spill that has reached surface water, Client or Arcadis must immediately notify the National Response Center (800-424-8802) in accordance with 40 CFR § 262.265. Other notifications to state and/or other local regulatory agencies may also be necessary.

Drilling Soil Cuttings and Muds

Soil cuttings are solid to semi-solid soils generated during trenching activities, subsurface soil sampling, or installation of monitoring wells. Depending on the drilling method, drilling fluids known as "muds" may be used to remove soil cuttings. Drilling fluids flushed from the borehole must be directed into a settling section of a mud pit. This allows reuse of the decanted fluids after removal of the settled sediments. Soil cuttings will be labeled and stored in 55-gallon drums with bolt-sealed lids.

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Excavated Solids

Excavated solids may include, but are not limited to: soil, fill, and construction and demolition debris. Prior to permitted treatment or offsite disposal, potentially hazardous excavated solids may be temporarily stockpiled onsite as long as the stockpile remains in the same AOC from where it was excavated. Potentially hazardous excavated solids removed from the AOC must be immediately containerized in labeled drums or closable top roll-offs lined with 9-mil polyvinyl chloride (PVC) sheeting and are subject to LQG storage time limits. Nonhazardous excavated solids can be stockpiled either inside or outside of the AOC, do not have to be containerized and are not subject to hazardous waste regulations. Potentially hazardous excavated solids must not be mixed with nonhazardous excavated solids. All classes of excavated solid stockpiles should be maintained in a secure area onsite. At a minimum, the floor of the stockpile area will be covered with a 20-mil high density polyethylene liner that is supported by a foundation or at least a 60-mil high density polyethylene liner that is not supported by a foundation. The excavated material will not contain free liquids. The owner/operator will provide controls for windblown dispersion, run-on control, and precipitation runoff. The run-on control system will prevent flow onto the active portion of the pile during peak discharge from at least a 25-year storm and the run-off management system will collect and control at least the water volume resulting from a 24-hour, 25-year storm (USEPA, 1992). Additionally, the stockpile area will be inspected on a weekly basis and after storm events. Individual states may require that the stockpile be inspected/certified by a licensed professional engineer. Stockpiled material will be covered with a 6-mil polyvinyl chloride (PVC) liner or sprayed dust control product. The stockpile cover will be secured in place with appropriate material (concrete blocks, weights, etc.) to prevent the movement of the cover.

Decontamination Solutions

Decontamination solutions are generated during the decontamination of personal protective equipment and sampling equipment. Decontamination solutions may range from detergents, organic solvents and acids used to decontaminate small field sampling equipment to steam cleaning rinsate used to wash heavy field equipment. These solutions are to be labeled and stored in closed head drums compatible with the decontamination solution. Decontamination procedures, including personnel and field sampling equipment, must comply with applicable Arcadis procedural documents.

Disposable Equipment

Disposable equipment includes personal protective equipment (e.g., tyvek coveralls, gloves, booties and APR cartridges) and disposable sampling equipment such as trowels or disposable bailers. If the media sampled exhibits hazardous characteristics per results of waste characterization sampling, contaminated disposable equipment will also be disposed of as a hazardous waste. If compatible with the original IDW waste stream (i.e., the IDW is a solid and the disposal equipment is a solid), the disposable equipment can be combined with the IDW. If these materials are not compatible (i.e., the IDW is a liquid and the disposal equipment is a solid), the disposable equipment will be stored onsite in separate labeled 55-gallon drums. Uncontaminated or decontaminated disposable equipment can be considered nonhazardous waste.

Purge Water

Purge water includes groundwater generated during well development, groundwater sampling, or aquifer testing. The volume of groundwater generated will dictate the appropriate storage procedure. Monitoring

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well development and groundwater sampling may generate three well volumes of groundwater or more. This volume will be stored in labeled 55-gallon drums. Aquifer tests may generate significantly greater volumes of groundwater depending on the well yield and the duration of the test. Therefore, large-volume portable polyethylene tanks will be considered for temporary storage pending groundwater-waste characterization.

Purged Water Storage Tank Decontamination and Removal

The following procedures will be used for inspection, cleaning, and offsite removal of storage tanks used for temporary storage of purge water. These procedures are intended to be used for rented portable tanks such as Baker Tanks or Rain for Rent containers. Storage tanks will be made of inert plastic materials. The major steps for preparing a rented tank for return to a vendor include characterizing the purge water, disposing of the purge water, decontaminating the tank, final tank inspection, and mobilization. Decontamination and inspection procedures are described in further detail below.

- Tank Cleaning: Most vendors require that tanks be free of any visible sediment and water before returning, a professional cleaning service may be required. Each specific vendor should be consulted concerning specific requirements for returning tanks.
- Tank Inspection: After emptying the tank, purged water storage tanks should be inspected for debris, chemical staining, and physical damage. The vendors require that tanks be returned in the original condition (i.e., free of sediment, staining and no physical damage).

8 WASTE MANAGEMENT

Soil/Solids Characterization

Waste characterization will be conducted in accordance with waste hauler, waste handling facility, and local/state/federal requirements. In general, RCRA hazardous wastes are those solid wastes determined by a Toxicity Characteristic Leaching Procedure (TCLP) test or to contain levels of certain toxic metals, pesticides, or other organic chemicals above specific applicable regulatory agency thresholds. If the one or more of 40 toxic compounds listed in Table I of 40 CFR § 261.24 are detected in the sample at levels above the maximum unregulated concentrations, the waste must be characterized as a toxic hazardous waste. Wastes can also be considered “listed” hazardous waste depending on site-specific processes.

Composite soil samples will be collected at a frequency of one sample per 250 cubic yard basis for stockpiled soil or one per 55-gallon drum per different waste stream for containerized. A four-point composite sample will be collected per 250 cubic yards of stockpiled material and for each drum waste stream. Sample and composite frequencies may be adjusted in accordance with the waste handling facility’s requirements and may be reduced for large volumes of waste with consistent properties. Waste characterization samples will be considered valid for consistent waste streams for a period of 1 year. Waste characterization samples may be analyzed for the TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA metals, and polychlorinated biphenyls (PCBs), as well as reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis. Site-specific constituents of concern including pesticides may require additional sampling. Please note that state- or local-specific regulations may require a different or additional sampling approaches.

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Wastewater Characterization

Waste characterization will be conducted in accordance with the requirements of the waste hauler, waste handling facility, and local/state/federal governments. In general, purge water should be analyzed by methods appropriate for the known contaminants, if any, that have been historically detected in the monitoring wells. Samples will be collected and analyzed in accordance with the requirements of the waste disposal facility. Wastewater characterization samples may be analyzed for TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA metals, and polychlorinated biphenyls, as well as corrosivity (pH), reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis. Site-specific constituents of concern including pesticides may require additional sampling. Please note that state- and/or local-specific regulations may require different or additional sampling approaches.

Sample Handling and Shipping

All samples will be appropriately labeled, packed, and shipped, and the chain-of-custody will be filled out in accordance with current Arcadis sample chain of custody, handling, packing, and shipping procedures and guidance instructions.

It should be noted that additional training is required for packaging and shipping of hazardous and/or dangerous materials. Please refer to the current Arcadis training requirements related to handling and shipping of samples, shipping determinations, and hazardous materials.

Preparing Waste Shipment Documentation (Hazardous and Nonhazardous)

Waste profiles will be prepared by the Arcadis CPM and forwarded, along with laboratory analytical data to the Client for approval/signature. The Client will then return the profile to Arcadis who will then forward to the waste removal contractor for preparation of a manifest. The manifest will be reviewed by Arcadis prior to forwarding to the Client for approval. Upon approval of the manifest, the Client will return the original signed manifest directly to the waste contractor or to the Arcadis CPM for forwarding to the waste contractor. Arcadis personnel may sign waste profiles and/or waste manifests on a case by case basis for clients, provided the appropriate agreement is in place between Arcadis and the client documenting that Arcadis is not the generator, but is acting as an authorized representative of the generator.

Final drum labeling and pickup will be supervised by an Arcadis representative who is trained and experienced with applicable waste labeling procedures. The Arcadis representative will have a copy of the drum inventory maintained in the field book and will reconcile the drum inventory with the profile numbers on the labels and on the manifest. Different profile numbers will be generated for different matrices or materials in the drums. For example, the profile number for drill cuttings will be different than the profile number for purge water. When there are multiple profiles it is critical that the proper label, with the profile number appropriate to a specific material be affixed to the proper drums. A copy of the Arcadis drum inventory will be provided to the waste transporter during drum pickup and to the facility receiving the waste.

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9 DATA RECORDING AND MANAGEMENT

Waste characterization sample handling, packing, and shipping procedures will be documented in accordance with relevant Arcadis procedures and guidance instructions as well as applicable client and/or project requirements, such as a Quality Assurance Project Plan or Sampling and Analysis Plan. Copies of the chain-of-custody forms will be maintained in the project file. Arcadis should photograph or maintain a copy of any hazardous waste manifest signed on behalf of Client in the corresponding office DOT record file.

10 QUALITY ASSURANCE

The CPM or APM will review all field documentation once per week for errors or omissions as compared to applicable project requirements including but not limited to: the proposal/scope of work, QAPP, SAP, HASP, etc. Deficiencies will be noted, tracked, and resolved. Upon correction, they will be noted for project documentation.

11 REFERENCES

United States Environmental Protection Agency (USEPA). 1992. Guide to Management of Investigation-Derived Wastes. Office of Remedial and Emergency Response. Hazardous Site Control Division. January 1992.

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ARCADIS Design & Consultancy for natural and built assets

TGI – Resource Conservation and Recovery Act (RCRA) Waste Characterization Sampling

Rev: 0

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Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
1	0	May 20, 2021	All	New TGI	Ryan Mattson

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Approval Signatures

Prepared by:		05/20/2021
	Ryan Mattson	Date:
Technical Expert Reviewed by:		05/21/2021
	Michelle Clayton	Date:

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1 Introduction

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 Scope and Application

The objective of this Technical Guidance Instruction (TGI) is to provide field sampling personnel procedures for collecting waste characterization samples from materials which are subject to the Resource Conservation and Recovery Act (RCRA) definition of solid waste at 40 CFR §261.2. Solid wastes, which include liquid, solid, or sludge – phase wastes, typically encountered by Arcadis are contaminated environmental media (soil and groundwater), contaminated building materials, drilling fluids, decontamination liquids, contaminated personal protective equipment (PPE), sorbent materials, construction and demolition debris, and disposable sampling materials. Arcadis may also be hired to sample client industrial or manufacturing wastes.

This TGI specifically excludes the following sampling which should be covered by more specific TGIs: polychlorinated biphenyl waste sampling under the Toxic Substances Control Act, radioactive waste sampling under the Atomic Energy Act, naturally occurring radioactive material or technologically enhanced naturally occurring radioactive material waste sampling, asbestos sampling, lead paint sampling, medical waste sampling, National Pollution Elimination Discharge permit sampling and publicly owned treatment works discharge sampling.

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Personnel utilizing this TGI should have a sampling plan, waste analysis plan or request that has been reviewed by a client or Arcadis hazardous waste Technical Expert and the sampler should understand location, scope, analytical testing laboratory, results timing, and data quality objectives for the particular sampling event prior to work. If this is not the case, stop work and contact the CPM for scope related questions or the hazardous waste Technical Expert for waste compliance questions. Arcadis hazardous waste Technical Expert advice is available by emailing regulatedwaste@arcadis.com.

This TGI describes the necessary equipment, field procedures, materials, regulatory references, and documentation procedures for waste characterization sampling. The procedures included in this TGI for waste characterizations sampling are based on the EPA's guidance document RCRA Waste Sampling Draft Technical Guidance (USEPA 2002). Waste characterization sampling provides data to help ensure the wastes are managed, transported and disposed in accordance with applicable federal, state, and local regulations. Although not comprehensive, the following laws and regulations pertaining to Hazardous Waste Management should be considered applicable. It is the Arcadis Certified Project Manager (CPM) and/or designated Technical Expert to determine which laws and regulations, at all levels of government, are applicable to each project site and activity falling under this TGI.

Federal Laws and Regulations

- Resource Conservation and Recovery Act (RCRA) 42 USC §6901-6987 (1976).
- Federal Hazardous Waste Regulations 40 CFR §260-261, Appendix I to §261, §264
- Department of Transportation (DOT) Hazardous Materials Transportation 49 CFR §171-173
- Occupational Safety and Health Administration (OSHA) Regulations 29 CFR §1910 and §1926

State Laws and Regulations

- To be determined based on location of site and location of treatment, storage, and/or disposal facility (TSDF) to be utilized.
- Regional, County, Municipal, and Local Regulations

This TGI may be modified by the CPM and/or Technical Expert for a specific project or client program, as required, dependent upon client requirements, site conditions, equipment limitations, or limitations imposed by the procedure. The resulting procedure employed to execute the work should be documented in the project work plans or reports. If changes to the sampling procedures are required due to unanticipated field conditions, the changes should be discussed with the CPM and/or Technical Expert as soon as practicable, and if approved to be performed, be documented.

3 Personnel Qualifications

Arcadis field sampling personnel will have current regulatory- and Arcadis-required health and safety training including 40-hour OSHA hazardous waste operations and emergency response (HAZWOPER) training, site supervisor training, site-specific training, first aid, and cardiopulmonary resuscitation (CPR), as needed. Personnel handling hazardous waste, including sampling, should have RCRA hazardous waste management training per 40 CFR §264.16. Additional state-specific hazardous waste management training may be required in certain states (i.e., California).

Arcadis personnel who ship samples that are subject to the definition of a DOT hazardous material (49 CFR 171.8) should have biennial DOT hazardous materials transportation training per 49 CFR § 172.704 (Arcadis

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requires every 2 years, DOT requires every 3 years). Arcadis field personnel should also comply with client-specific training. In addition, Arcadis field sampling personnel should be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The project health and safety plan (HASP) and other documents may identify other training requirements or access control requirements.

4 Equipment List

The following equipment and materials, as required, should be available for waste characterization sampling:

All sampling events:

- Appropriate personal protective equipment as specified in the HASP
- Leather gloves
- Nitrile gloves
- Appropriate labeling, packing, chain-of-custody forms, and shipping materials as determined by the CPM and/or Technical Expert.
- Decontamination equipment/tools including Alconox®/Liquinox®-band detergent spray bottle, deionized or distilled water spray bottle, brushes, buckets.
- Indelible ink and/or permanent marking pens
- Appropriate sample containers, labels, and forms
- Digital camera
- Field logbook

Additional equipment/materials for Container Sampling – Solid Phase Waste

- Hammer
- 5/8” drum wrench/socket
- Stainless steel or disposable plastic hand spade
- Stainless steel scoop
- Stainless steel bucket auger

Additional equipment/materials for Container Sampling – Liquid Phase Waste

- Hammer
- 5/8” drum wrench/socket
- Step stool or ladder
- Disposable bailer, coliwasa sampler, or dipper

Additional equipment/materials for Process Waste Streams

- Process diagram
- Lockout/tagout information
- Step stool or ladder
- Stainless steel or disposable plastic hand spade
- Stainless steel scoop
- Disposable bailer, coliwasa sampler, or dipper

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Additional equipment/materials for Uncontained Release Area

- Air monitoring equipment
- Underground utilities clearance checklist
- Long measuring tape or wheel
- Pin flags
- Stainless steel or disposable plastic hand spade
- Stainless steel scoop
- Stainless steel bucket auger

Additional equipment/materials for Destructive Building Material Sampling

- Plastic sheeting
- Hammer drill with appropriate bits
- Hammer
- Flathead screwdriver
- Chisel
- Putty knife
- Generator or other power source
- Zip seal plastic bags
- Paper plates

5 Cautions

This TGI does not provide procedures for sampling waste streams where there is no knowledge of waste origin or potential hazards. If such a situation arises, the sampler should stop work and consult the CPM and a Technical Expert.

6 Health and Safety Considerations

As determined by the site's known and suspected hazards, appropriate PPE should be worn by all field personnel within the designated work area. Exposure air monitoring may be required during certain field activities as stated in the Site HASP.

Arcadis field personnel and subcontractors will be trained in and perform their work in compliance with all applicable federal, state, and local health and safety regulations as well as Arcadis' HASP and applicable Client health and safety requirements.

Filled waste containers can be very heavy, become unbalanced, and/or spill contents. Waste containers stored for extended periods of time may be subject to deterioration. Personnel should attempt to sample waste containers without moving them and, if necessary, use appropriate moving techniques and equipment for safe handling. Container lids must be replaced after sampling to maintain RCRA compliance. Large containers such as rolloff bins and frac tanks may require climbing to access sampling ports. A second sampler is recommended as a spotter for such sampling events. Sampling wastes from active processes can involve opening valves or accessing confined spaces. Stop work and communicate with the CPM and process operator if there is any misunderstanding or change in scope of sampling. Destructive bulk sampling can generate projectiles, please make sure the area is clear of ancillary personnel and upgrade PPE as appropriate. Destructive sampling can

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also encounter hidden utilities. Sampling of uncontained wastes may result in exposure to airborne contamination or if the waste is in stockpiles may involve gravity hazards. Upgrade monitoring equipment and plan work in advance to minimize these hazards.

7 Procedure

Sampling Methodology

RCRA requires that waste characterization samples be representative of the waste stream. A representative sample is defined in 40 CFR 260.10 as a sample of a universe or a whole that can be expected to exhibit the average properties of the universe or whole. Arcadis personnel should work with the client, waste Technical Expert and disposal facility on an approach to collect representative waste characterization samples and prepare them for shipment for laboratory analysis. The laboratory used for waste characterization analysis should have the appropriate federal and state accreditations and may be required to be pre-approved by the Client.

Sampling Containerized Solid Wastes

1. Assess the integrity of the container for labels, bulging, leaks. If the container appears compromised or unlabeled, stop work and contact the CPM.
2. Open the container using appropriate tools.
3. Record visual appearance and volume of waste in field logbook.
4. Collect volatile constituent samples (volatile organic compounds, total petroleum hydrocarbons – gasoline range, etc.) as grab samples at a random location using a method to minimize the loss of volatiles, typically directly into laboratory provided sampling devices/containers.
5. Collect semi and nonvolatile constituent samples (semi-volatile organic compounds, etc.) as grab samples for 4-point composites from random locations in the accessible waste layer. The nonvolatile sample compositing should be performed by the laboratory. A disposable or stainless-steel sampling tool or bucket auger may be used if deep container sampling is requested. Decontaminate any tool used between sampling different locations.
6. Place samples in laboratory provided sample containers.
7. Close the container by replacing all components disassembled to open it.
8. Take a photograph of the container.
9. Note the container sampled, photo number and sample ID in the field logbook.

Sampling Containerized Liquid Wastes

1. Assess the integrity of the container for labels, bulging, leaks. If the container appears compromised or unlabeled, stop work and contact the CPM.
2. If the container is a tank, sampling from a hatch or opening at the top is preferred over a valve at the bottom because of potential waste stratification.
3. Open the container using appropriate tools.
4. Record visual appearance and volume of waste in field logbook and assess the waste for separate phases (non-aqueous phase liquid [napl], liquid, sediment) and record percentages by volume of each in field logbook. If there are separate phases, contact CPM to advise if discrete sampling of each phase is required.
5. Open an unused disposable liquid sampling tool (such as a bailer, coliwasa or dipper). If discrete phase sampling is required, choose a sampling tool that is capable of collecting the desired phase only and

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insert the sampling end of the tool in the phase to be sampled. If discrete phase sampling is not required, choose a sampling tool, and stir the waste.

6. Collect a representative sample of the waste using the tool into laboratory provided sample containers.
7. If pH is requested by field test, apply some waste from the liquid sampler to pH paper/pH meter and photograph the paper result or equipment display screen showing the pH value for project records. Note if using pH paper, it must be capable of reading pH in the range of at least 1 to 13 pH units, with a larger range acceptable.
8. Close the container by replacing all components disassembled to open it.
9. Take a photograph of the container.
10. Note the container sampled, photo number and sample ID in the field logbook.

Sampling Process Waste Streams

Process waste streams typically have designated sample collection locations (built in sampling ports, covered pits, sumps etc.) to allow collection of waste characterization samples. The sampler should consult the process operator and receive authorization before collecting any samples from an active process.

1. Record the name of the sampling location in field notes and on sample labels.
2. Collect a sample directly from the sampling location into the laboratory-provided container, if possible. Some liquid processes may need to be sampled using a dipper which should be passed through the process waste in one continuous motion to collect the sample and transfer to laboratory-provided containers. A disposable flat scoop sampling tool can be used to collect solid process wastes off conveyors, etc. prior to transferring to laboratory-provided containers.
3. Take a photograph of the sample location.
4. Note the sample location, photo number and sample ID in the field logbook.

Sampling Uncontained Release Areas

Sampling of an uncontained release or spill area can be performed as follows:

New Releases:

1. Visually assess the horizontal boundaries of the spill. The first round of waste characterization is typically biased by collecting waste characterization samples that are most likely to be “worst case”, i.e., visually impacted and near the center of the spill. The samples should be collected as grab samples with ideally a minimum of four (4) grab samples to characterize a new waste stream to statistical significance.
2. Collect volatile constituent samples (volatile organic compounds, total petroleum hydrocarbons – gasoline range, etc.) semi and nonvolatile constituent samples (semi-volatile organic compounds, etc.) as grab samples centered on the release location. A disposable or stainless-steel sampling tool may be used or a bucket auger if deep sampling is requested. Volatile constituent samples should be collected using a method to minimize the loss of volatiles, typically directly into laboratory provided sampling devices/containers. Decontaminate any tool used between sampling different locations.
3. Place samples in laboratory provided sample containers.
4. Take a photograph of the sampling location.
5. Note the sampling location, photo number and sample ID in the field logbook.

Stockpiles:

1. Assess the size of the stockpile and work with the client, waste Technical Expert and disposal facility on an approach to collect an appropriate number of representative samples.

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2. Collect volatile constituent samples (volatile organic compounds, total petroleum hydrocarbons – gasoline range, etc.) semi and nonvolatile constituent samples (semi-volatile organic compounds, etc.) as grab samples from randomly selected places in the stockpile. A disposable or stainless-steel sampling tool may be used for samples near the outer edge of the stockpile and a bucket auger can be used for sampling deeper into the stockpile. Volatile constituent samples should be collected from stockpile material inside the outer "shell" of the stockpile that has had less exposure to air for using a method to minimize the loss of volatiles, typically directly into laboratory provided sampling devices/containers. Decontaminate any tool used between sampling different locations.
3. Collect semi and nonvolatile constituent samples (semi-volatile organic compounds, etc.) as either grab samples from randomly selected places in the stockpile, if characterization samples will be used to separate the stockpile into different waste streams, or as grab samples for 4-point composites from random locations, if the stockpile will be disposed collectively as one waste stream. Nonvolatile sample compositing should be performed by the laboratory. A disposable or stainless-steel sampling tool may be used for samples near the outer edge of the stockpile and a bucket auger can be used for sampling deeper into the stockpile. Decontaminate any tool used between sampling different locations.
4. Place samples in laboratory provided sample containers.
5. Take a photograph of the sampling location.
6. Note the sampling location, photo number and sample ID in the field logbook.

Historical Releases:

Waste characterization for historical release areas subject to future remediation is typically performed in accordance with a site investigation work plan and is out of scope for this TGI.

Destructive Sampling Building Materials

1. Assess the building material and area to be sampled. Surface coatings and softer material such as sheet rock can be sampled with hand tools (hammer, chisel, putty knife, etc.). Harder materials such as wood, masonry and concrete may require a hammer drill with an appropriate bit. Confirm the volume/mass requirements for the sample with the analytical lab to collect a sufficient amount of sample for analysis while minimizing damage to the sample area.
2. Sampling Vertical Surfaces:
    - Place plastic sheeting under the sampling area.
    - If directed to collect a surface coating sample, scrape material into a labeled laboratory provided sampling container or, if necessary, a new plastic bag beneath the sampling area.
    - If directed to collect a representative sample of the entire building material, drill into the full thickness of the material to be disposed, collecting shavings on a labeled new paper plate beneath the sampling area. Move to an adjacent area and drill another full thickness hole if additional sampling volume/mass is needed.
3. Sampling Vertical Surfaces:
    - Place plastic sheeting around the sampling area.
    - If directed to collect a surface coating sample, scrape material, and transfer it to a labeled laboratory provided sampling container.
    - If directed to collect a representative sample of the entire building material, place a labeled new paper plate directly over the sampling area and drill through the plate into the full thickness of the material to be disposed. Shavings should collect on the paper plate. Move to an adjacent area

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and drill another full thickness hole using a new paper plate each time if additional sampling volume/mass is needed.

4. Record visual appearance and volume of waste in field logbook.
5. Transfer samples from bags or plates to laboratory provided sample containers.
6. Take a photograph of the sampling location.
7. Note the sampling location, photo number and sample ID in the field logbook.

Sampling Frequency

The sampler should work with the client, waste Technical Expert and disposal facility on an approach to collect an appropriate number of representative samples. In the absence of any other frequency directive, waste characterization samples should be collected at the following frequencies:

- Large Volume Soil/Sludge Waste Steams (Uncontained, Stockpiles, Rolloff Bins, etc.): minimum one representative sample per 200 cubic yards.
- Large Volume Liquid Waste Streams (Tanks, Ponds, etc.): minimum one representative sample per waste stream.
- Small Volume Soil/Sludge Waste Streams (Drums, Totes, etc.):
    - 1-4 containers: one grab sample from one randomly selected container for volatile constituents; grab samples from each container to be combined by laboratory into 4-point composite for nonvolatile constituents.
    - More than 4 containers: grab samples for volatile constituents from randomly selected containers representing 20% of total number of containers; grab samples for nonvolatile constituents from the same 20% of total number of containers to be combined by laboratory into 4-point (or less) composites.
- Small Volume Liquid Waste Streams (Drums, Totes, etc.): minimum one representative sample from randomly selected containers representing 20% of total number of containers.

Waste Characterization Sample Analytical Parameters

Waste characterization should be conducted in accordance with the requirements of the client, waste handling facility, and local/state/federal governments. In general, wastewater should be analyzed by methods appropriate for the known or suspected contaminants in the waste stream. Solid phase waste characterization samples may be analyzed for toxicity characteristic leaching procedure (TCLP) VOCs, TCLP SVOCs, TCLP RCRA metals, and PCBs, as well as reactivity. Liquid and sludge phase waste characterization samples may be analyzed for TCLP VOCs, TCLP SVOCs, TCLP RCRA metals, and PCBs, as well as corrosivity (pH), reactivity and flammability (flashpoint). Project-specific constituents of concern including PCBs, herbicides and pesticides may require additional sampling. Please note that state- or local-specific regulations may require a different or additional sampling approaches such as total petroleum hydrocarbon sampling, fish bioassay sampling, etc.

Sample Handling and Shipping

All samples should be appropriately labeled, packed, and shipped in accordance with Arcadis sample cooler packing procedure/video available at the Arcadis Transportation Safety SharePoint site. The chain-of-custody should be filled out in accordance with current Arcadis sample chain of custody standard operating procedure (SOP) available from the Arcadis Virtual Library. Any sample compositing instructions for the laboratory should be included on the chain of custody. The Arcadis shipping determination form (Arcadis Transportation Safety SharePoint) should be completed prior to the first shipment of samples from a project site.

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As noted under the qualifications section above, biennial training is required by Arcadis for packaging and shipping of hazardous and/or dangerous materials. Please refer to the current Arcadis training requirements related to handling and shipping of samples, shipping determinations, and hazardous materials.

8 Data Recording and Management

Waste characterization sample handling, packing, and shipping procedures shold be documented in accordance with relevant Arcadis procedures and guidance instructions as well as applicable client and/or project requirements, such as a Quality Assurance Project Plan, Sampling and Analysis Plan, or Waste Analysis Plan. Copies of the chain-of-custody forms should be maintained in the project file. Arcadis should photograph or maintain a copy of any hazardous waste manifest signed on behalf of Client in the corresponding office DOT record file.

9 Quality Assurance

The CPM should review all field documentation once per week for errors or omissions as compared to applicable project requirements including but not limited to the proposal/scope of work, QAPP, SAP, WAP, HASP, etc. Deficiencies should be noted, tracked, and resolved. Upon correction, they should be noted for project documentation.

10 References

United States Environmental Protection Agency (USEPA). 2002. RCRA Waste Sampling Draft Technical Guidance. Office of Solid Waste. August.

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TGI – Sample Chain of Custody

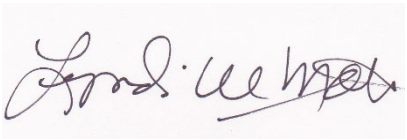
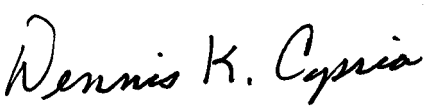
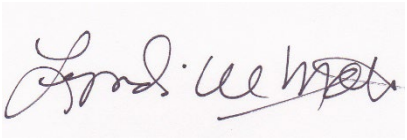
Rev: 3

Rev Date: March 28, 2022

Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
	0	April 19, 2017	All	Re-write to COC only	Richard Murphy
	1	May 23, 2017	4,7,9	Add: Guidance on use of previous version of TGI. Add: Info on COCs for multiple shipping containers Modify: Move letter i. to letter m. and change to “when appropriate”	Peter Frederick
	2	April 29, 2020	4, 11	Remove obsolete link	Lyndi Mott
	3	December 28, 2022	All	Updated Arcadis format Added to 6c. Collection time between COC and container must match. Added to 6o. Add name of overnight courier when relinquishing samples. Updated reference documents and added internet links.	Lyndi Mott

Approval Signatures

Prepared by:		3/28/2022
	Lyndi Mott (Preparer)	Date
Reviewed by:		3/28/2022
	Dennis Capria (Chain of Custody Reviewer)	Date
Reviewed by:		12/22/2021
	Lyndi Mott (Subject Matter Expert)	Date

1 Introduction

This Technical Guidance Instruction (TGI) provides the procedure for Arcadis field personnel for required documentation during the collection of environmental field samples and transfer of custody to a laboratory. It provides direction for completion of the Chain of Custody form that must accompany collected field samples for analysis by a laboratory.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

This TGI describes the general Chain of Custody (COC) procedures and guidance instructions for samples collected from project sites that are relinquished from Arcadis' possession.

COC is defined as the maintenance of an unbroken record of possession of an item from the time of its collection through some analytical or testing procedure. COC is typically documented by a written record of the collection, possession, and handling of samples collected from a project location. Each sample will be tracked by a documented record that efficiently documents the individuals who were responsible for the sample during each successive transfer of that sample to various recipients beyond Arcadis' possession. This information can be used to legally establish the integrity of the samples and therefore the analytical results derived from the samples. This

information can be used in addition to other records and documentation regarding the samples, such as field forms, field logs, and photographs.

A sample is considered under custody if:

- It is in your possession; or
- It is in your view, after being in your possession; or
- It was in your possession and then you then locked it up to prevent tampering; or
- It is in a designated secure area.

Continued use of previous version of TGI:

Although not recommended, Arcadis program-, project-, and client-teams may be able to use the previous version of this TGI provided that it meets all of the quality expectations of Arcadis and client and meets applicable regulatory requirements. It is up to the program, project, and/or client-team leader to determine whether it is appropriate to adopt the current TGI or to continue using the previous version.

However, all new work not associated with the previous version of this TGI must be performed with the current version of the TGI.

When adopting this new TGI, users of the previous versions must be aware that specific handling, packing, and shipping procedures and guidance has been removed and that those should be addressed within program or project plans (e.g., Quality Assurance Project Plans (QAPP), Work Plans, Sampling and Analysis Plans (SAPs), etc.) or in a more detailed TGI specific to that sampling activity, whether related to media, constituent/analyte, client, state, etc.

In addition, adopting this new TGI will require users to refer to the Arcadis Department of Transportation (DOT) Safety Program for procedures and guidance on the determination and handling, packing, and shipping of samples that are or may be considered hazardous materials.

4 Personnel Qualifications

Arcadis personnel performing work under the purview of this TGI will have received appropriate training and have field experience regarding the collection of samples from project locations. Arcadis personnel will have all other applicable and appropriate training relevant to the sampling work and project site.

5 Equipment List

The following list provides materials that may be required for each COC. Project reporting and documentation requirements must be reviewed with the CPM prior to execution of work. Additional materials, tools, equipment, etc. may be required, and project staff are required to verify with the CPM and/or Technical Expert what specific equipment is required to complete the COC.

- Indelible ink pen (preferably either black or blue ink);
- COC form **(Appendix A)** from either Arcadis, laboratory receiving and analyzing the samples, or other applicable and appropriate entity for the work performed;
- When appropriate, such as for litigation or expert testimony work, custody seals or tape.

6 Cautions

One way in which the law tries to ensure the integrity of evidence is by requiring proof of the chain of custody by the party who is seeking to introduce a particular piece of evidence.

A proper chain of custody requires three types of affirmations: (1) affirmation that a sample is what it purports to be (for example, soil collected from a specified location and depth); (2) affirmation of continuous possession by each individual who has had possession of the sample from the time it is collected until the time it is analyzed or held by a laboratory; and (3) affirmation by each person who has had possession that sample remained in substantially the same condition and not contaminated or affected by outside influences from the moment one person took possession until the moment that person released the evidence into the custody of another (for example, affirmation that the sample was stored in a secure location where no one but the person in custody had access to it).

Proving chain of custody is necessary to "lay a foundation" for the samples in question, by showing the absence of alteration, substitution, or change of condition.

Ensure that appropriate sample containers with applicable preservatives, coolers, and packing material are planned for and provided at the site at the time of sample collection.

Understand the offsite transfer requirements of the samples for the facility at which samples are collected.

If overnight courier service is required schedule pick-up or know where the drop-off service center is located and the hours of operation.

An Arcadis employee appropriately trained at the correct level of internal hazardous materials/DOT)shipping must complete an Arcadis shipping determination to address applicable DOT and International Air Transport Association (IATA) shipping requirements. Review the applicable Arcadis procedures and guidance instructions for sample packaging, and labeling. Prior to using air transportation, confirm air shipment is acceptable under DOT and IATA regulations.

The person relinquishing possession of the samples or other member of the project team should contact the final recipient of the samples to confirm receipt and review any special provisions on the COC or questions that they may have.

7 Health and Safety Considerations

Follow the health and safety procedures outlined in the project/site Health and Safety Plan (HASP) as well as other applicable H&S requirements, such as:

- Arcadis Hazardous Material/DOT handling, packaging, and shipping training
- Project site-specific H&S training
- Client-specific H&S training
- Constituent-specific H&S training
- Media-specific H&S training

8 Procedure

Collected samples must be uniquely identified, and properly documented, containerized, labeled with unique identifier, possessed in a secure manner during remainder of sampling event, packaged, and shipped to recipient laboratory.

Sample Identification

The method of sample identification depends on the type of measurement or analyses performed. In some cases, in-situ measurements of existing conditions and/or sample location must be made during sample collection.

These data will be recorded directly on field forms, logbooks, or other project record data sheets used to permanently retain this information for the project file. Examples of location identification information includes: latitude/longitudinal measurements, compass directions, well number, building number, floor number, room name, or proximity to a site feature unique to the site. Examples of in-situ measurements are pH, temperature, conductivity, flow measurement, or physical condition of the media being sampled. Physical samples collected are identified by a unique identifying number or code on a sample tag or label. These physical samples are removed from the sample location and transported to a laboratory for analyses.

In some cases, before samples are placed into individual containers and labeled as individual samples, samples may be separated into portions depending upon the analytical methods and required duplicate or triplicate analyses to be performed.

When completing a COC for samples, personnel must complete the following:

1. Written COCs must be completed with indelible ink (preferably either black or blue colored ink).
2. Written COCs must be completed using legible printed writing, and not cursive writing.
3. All entry fields on the COC form must be completed. If information is not applicable for a specific entry field, personnel will either put “N/A” or use a strike-out line or dash like “----------“ to indicate no applicable information is needed for that field.
4. Use of quotation marks or lines/down arrows to represent repetitive/duplicative text in similar fields.
5. Regardless of the type or specific COC form, the following pertinent information must be provided on the COC form:
    a. Arcadis project number
    b. Arcadis project name
    c. Project location, including street address, city, state, building number, providing as much detail as appropriate
    d. Recipient laboratory contact and sample receiving shipping location information
    e. Entities’/persons’ contact information for who will be receiving analytical results
    f. Name of sampler, i.e., person collecting sample and relinquishing possession of samples to the next entity in the chain of custody
    g. Date of sample collection
    h. If appropriate for the sample media, contaminant/constituent of concern, or analytical method, document time of sample collection using standard military time
    i. Sample analytical method(s)

j. Turnaround time required for analyses and/or reporting

k. Instructions to laboratory regarding handling, timing, analyses, etc. as applicable and appropriate.

l. Printed name and signature of the individual person who collected the samples and relinquishing possession of the samples

m. If appropriate or when documentation of the specific sample collection method will influence how the laboratory handles, prepares, or analyzes the samples, document the sample collection methodology used for collecting the samples (e.g., ASTM D5755)

6. The following additional specific information will be entered on the COC form, regardless of what type of COC is being used:

a. Unique Sample Identifier – The sample identifier (ID) must be unique to the individual sample it is applied to. The information in which the sample ID conveys is determined by the CPM, Technical Expert, and/or other project team members in advance of sample collection so that sample identification is consistently applied for the project. The sample nomenclature may be dictated by a specific client, program, or project database and require unique identification for each sample collected for the project. Consult with the CPM and/or Technical Expert for additional information regarding sample identification.

The sample ID could convey specific information regarding the sample to aid personnel in recognizing what the sample represents, or they may be arbitrary so as to facilitate the anonymity of the sample location, media, constituent of concern, project site, etc.

Examples of unique identifiers include:

1. Well locations, grid points, or soil boring identification numbers (e.g., MW-3, X-20, SB-30). When the depth interval is included, the complete sample ID would be “SB-30 (0.5-1.0) where the depth interval is in feet. Please note it is very important that the use of hyphens in sample names and depth units (i.e., feet or inches) remain consistent for all samples entered on the chain of custody form. DO NOT use the apostrophe or quotes in the sample ID.

2. Sample names may also use the abbreviations “FB,” “TB,” “FD” and “DUP” as prefixes or suffixes to indicate that the sample is a field blank, trip blank, or field duplicate, respectively.

b. List the date of sample collection. All indicated dates must be formatted using either mm/dd/yy (e.g., 03/07/09) or mm/dd/yyyy (e.g., 03/07/2009).

c. List the local time that the sample was collected. The time value should be presented using military format. For example, 3:15 P.M. should be entered as 15:15. The time listed on the COC form must match the sample collection time on the sample container(s).

d. Samples should be indicated to be either “Grab” or “Composite”. Grab samples are collected from only one unique location at one specific point in time.

e. Composite samples are a group of individual samples that are combined for analysis in their totality. Composite samples need to be documented if they are either collected from a number of different locations over a broader area to be representative of the entire area being sampled, or if they are representative of a single location over an extended period of time.

f. If used, preservatives for the individual sample will be noted.

g. The requested analytical method(s) that the samples are being analyzed for must be indicated. As much detail, as necessary, should be presented to allow the analytical laboratory to properly analyze the samples. For example, polychlorinated biphenyl (PCB) analyses may be represented by entering "EPA Method 8082 – PCBs" or "EPA PLM 600-R93-116." In cases where multiple analytical methods and/or analytical parameters are required for an individual sample, each method should be indicated for the sample (e.g., EPA 8082/8260/8270 or EPA PLM/400-point count).

h. If there are project-specific sample analytes to be reported, they should be specifically listed for each individual sample (e.g., 40 CFR 264 Appendix IX).

i. The total number of containers for each analytical method requested should be documented. This information may be included under the parameter or as a total for the sample.

j. When necessary, note which samples should be used for site specific matrix spikes in the Remarks or Comments field.

k. Indicate special project-specific requirements pertinent to the handling, shipping, or analyses. These requirements may be on a per sample basis such as "extract and hold sample until notified," or may be used to inform the laboratory of special reporting requirements for the entire sample delivery group (SDG).

l. Indicate turnaround time (TAT) required for samples on COC. If individual samples have differing TATs, the different TATs for each sample or groups of samples must be clearly indicated.

m. Provide contact name and phone number in the event that problems are encountered when samples are received at the laboratory. The person relinquishing possession of the samples or other member of the project team should contact the final recipient of the samples to confirm receipt and review any special provisions on the COC or questions that they may have.

n. If available, attach the Laboratory Task Order or Work Authorization forms.

o. The "Relinquished By" field must contain the signature of the Arcadis person who relinquished custody of the samples to the next entity in the chain of custody, which may be another person, the shipping courier, or the analytical laboratory. If a courier, enter the shipping courier in the "Received by" such as FedEx. The date/time relinquished should be when the person signs the COC and seals the cooler or shipping container for pick-up by the shipping courier.

p. Dates and times must be indicated using the following format:

   1) Date: either mm/dd/yy e.g., 01/01/17 OR mm/dd/yyyy e.g., 01/01/2017

   2) Time: use military format, e.g., 9:30 a.m. is 0930 and 9:30 p.m. is 2130

q. The "Received By" section is signed by sample courier or laboratory representative who received the samples from the sampler. The laboratory will sign upon laboratory receipt from the overnight courier service.

7. When more than one page of the COC form is required to complete the total number of samples, use as many sheets as necessary to accurately and clearly, document the samples and information. Some COCs may have a standard first page/cover page, and subsequent pages may not contain all the detailed fields as

the first page/cover page. Ensure that any subsequent pages convey all of the necessary and pertinent information for each individual sample as required in this procedure document.

8. Pages of the COC must retain a page count of the total number of pages; e.g., Page 1 of 3, Page 2 of 3, Page 3 of 3.
9. Upon completing the COC forms, forward the original signed COC with the sample package. Ensure that the original COC form is secured with the sample package so that it remains with the physical samples for the duration of transport and handling to its final destination and ensure that the COC form will not be become damaged or rendered unreadable due to sample breakage/leakage if stored inside the sample shipping container or outside influences if COC is stored in an outside plastic pouch to the container.
10. If you've collected enough samples that would require more than one container to ship them all to the same laboratory or location, then each separate/individual container that contains any number of samples must have a separate COC representing only those samples contained within that specific container. For example, if you have 3 total shipping containers for all of your samples, you must have a total of 3 separate, individual COCs for each of the 3 containers representing only those samples in their representative container. Thus, every container holding samples must have its own, individual COC.
11. If electronic chain of custody (eCOC) forms are utilized, ensure that the requirements of this procedure and guidance instructions are followed to the extent possible. Verify that proper signature and COC procedures are maintained with the CPM and/or Technical Expert when using eCOC.

9 Waste Management

Not Applicable.

10 Data Recording and Management

The original signed COC shall be submitted with the samples. Copies of COC records will be transmitted to the CPM or designee at the end of each day unless otherwise directed by the CPM. The sampling team leader retains copies of the chain of custody forms for filing in the project file. Record retention shall be in accordance with client- and project-specific requirements and Arcadis policies, the most stringent will apply.

The option to use the Electronic Chain of Custody (eCOC) form in conjunction with the appropriate sample application(s) may be available through the FieldNow® program but is currently limited to a select list of approved analytical laboratories. Use of the eCOC application is intended to reduce common transcription errors both by field staff and laboratory staff on a conventional handwritten paper COC. Once the eCOC form is completed and approved on the field tablet by field staff, a PDF version of the form is automatically emailed to each assigned team member. In addition, a dedicated or mobile printer is recommended for printing a hard copy of the completed eCOC to be included in each sample cooler to meet laboratory requirements.

11 Quality Assurance

COC forms will be legibly completed in accordance with this procedure and guidance instruction document, as well as other applicable and appropriate project documents such as SAP, Quality QAPP, Work Plan, or other project guidance documents.

COC records will be reviewed by the CPM or their appropriate designee for completeness and accuracy to the applicable requirements. Non-conformances will be noted and corrected in a timely manner on the copies retained by Arcadis as well as contacting the ultimate receiving entity for correction to the originally signed COC in their possession.

12 References

Arcadis Transportation Safety Program requirements, procedures, and guidance instructions.

EPA Samplers' Guide – Contract Laboratory Program Guidance for Field Samplers, EPA document EPA-540-R014-013 October 2014 https://www.epa.gov/sites/default/files/2015-03/documents/samplers_guide.pdf.

EPA Region III – Sample Submission Procedures for the Office of Analytical Services and Quality Assurance (OASQA) Laboratory Branch revision 14.0 October 18, 2018, https://www.epa.gov/sites/default/files/2018-12/documents/sample-submission-procedures-rev14.pdf.

EPA Region IV Science and Ecosystem Support Division Operating Procedure for Sample and Evidence Management May 25, 2016, https://www.epa.gov/sites/default/files/2015-06/documents/Sample-and-Evidence-Management.pdf.

Attachment A

Chain of Custody and Laboratory Analysis Request Form

ARCADIS

ID#

CHAIN OF CUSTODY & LABORATORY ANALYSIS REQUEST FORM

Page ____ of ____

Lab Work Order #

Send Results to:

Contact & Company Name:

Telephone:

Address:

Fax:

City State Zip

E-mail Address:

Project Name/Location (City, State):

Project #:

Sampler's Printed Name:

Sampler's Signature

SAMPLE ID	Collection Date	Collection Time	Type (✓) Comp	Type (✓) Grab	Matrix

Preservative									
Filtered (✓)									
# of Containers									
Container Information									
PARAMETER ANALYSIS & METHOD									
									REMARKS

Keys

Preservation Key:
A. H_2SO_4
B. HCL
C. HNO_3
D. NaOH
E. None
F. Other: _____
G. Other: _____
H. Other: _____

Containment Information Key
1. 40 ml Vial
2. 1 L Amber
3. 250 ml Plastic
4. 500 ml Plastic
5. Encore
6. 2 oz. Glass
7. 4 oz. Glass
8. 8 oz. Glass
9. Other: _____
10. Other: _____

Matrix Key:
SO - Soil
W - Water
T - Tissue
SE - Sediment
SL - Sludge
A - Air
NL - NAPL/Oil
SW - Sample Wipe
Other: _____

Special Instructions/Comments

☐ Special QA/QC Instructions (✓)

Laboratory Information and Receipt		Relinquished By	Received By	Relinquished By	Laboratory Received By
Last Name:	Cooler Custody Seal (✓)	Printed Name:	Printed Name:	Printed Name:	Printed Name
☐ Cooler packed with ice (✓)	☐ Intact ☐ Not Intact	Signature:	Signature:	Signature:	Signature:
Specify Turnaround Requirements:	Sample Receipt	Firm:	Firm:	Firm:	Firm:
Shipping Tracking #:	Condition/Cooler Temp:_____	Date/Time:	Date/Time:	Date/Time:	Date/Time:

SOP – Sample Chain of Custody Rev1_May 23, 2017

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TGI - In-Situ and Ex-Situ Water Quality Parameters – Surface Water and Groundwater

Rev: 1

Rev Date: April 15, 2022

Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
	0	October 16, 2018	All	Updated and re-written as TGI (in-situ and ex-situ adaptation)	Marc Killingstad
	1	April 15, 2022	All	Review and update to include surface water turbidity sensors	Shannon Dunn / Marc Killingstad

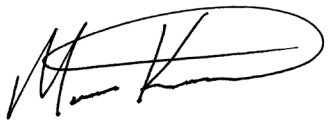
Approval Signatures

Prepared by: 4/15/2022



Chris Shepherd, PG — Date

Reviewed by: 4/15/2022



Marc Killingstad (Subject Matter Expert) — Date

1 Introduction

This Technical Guidance Instruction (TGI) describes methods used to measure water quality parameters in surface water as well as groundwater both in-situ and ex-situ.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

Water quality parameters (e.g., turbidity, specific conductivity, pH, and temperature) of natural waters are usually measured in the field. The temperature, pH, specific conductivity, dissolved oxygen (DO), and oxidation reduction potential (ORP) and turbidity of water will be measured in-situ or ex-situ with amultiparameter water quality meter (i.e., multimeter).

Conductivity is the ability of a solution to pass an electric current. This current is carried by inorganic dissolved solids. The measurement is useful to quantify the chemical purity of the water relative to the amount of dissolved solids in a solution. Generally, temperature-compensated conductivity, termed specific conductivity, is measured by a water quality meter. Specific conductivity can also be used to estimate total dissolved solids (TDS) in solution

(grams per liter or g/L) by multiplying the specific conductivity (measured in milli-siemens per centimeter or mS/cm) by 0.65 or a matrix specific factor, which assumes that sodiumchloride is a reasonable surrogate for the dissolved solids in solution.

Measuring the concentration of DO in water is an important component in evaluating the quality of naturalas well as contaminated waters. The effects of wastes on rivers/streams, the suitability of water for fish and other organisms, as well as the effects of remediation efforts, can often be ascertained from the DO content. Dissolved oxygen levels generally range from 5 to 9 milligrams per liter (mg/L) when measured inwater that is in equilibrium with air, depending on the temperature and barometric pressure; however, levels may be lower in a 'reducing' environment where anoxic conditions exist in water (e.g., gasoline plume undergoing bioremediation). DO is measure in both mg/L and percent.

The measurement from an ORP sensor represents the net status of all the oxidation and reduction reactions in the sample being measured. Positive results indicate an oxidizing environment while negativeresults indicate a reducing environment. ORP is determined by measuring the potential of a chemically- inert electrode which is immersed in the solution. The sensing electrode potential is read relative to the reference electrode of the pH probe and the value is presented in millivolts (mV).

ORP meter results can indicate possible contamination and can be particularly useful if it is known that one component of the sample is primarily responsible for the observed value. For example, excess chlorine in a sample will result in a large positive ORP value while the presence of hydrogen sulfide willresult in a large negative ORP value. Together with pH, temperature, and knowledge of the dominant species in a sample, ORP results help predict the oxidation state of ions in solution and whether certainreactions may take place.

Temperature readings will be taken at each water sampling location to assist understanding the water quality as well as compensating measurements. A thermometer is typically part of the multimeter. If not,a temperature probe or thermometer will be used.

Turbidity is a measure of the relative clarity of water. It is an optical measurement of the amount of light scattered in the water. Turbidity is often used as a proxy for the amount of suspended particles in the water. Turbidity is typically reported in nephelometric turbidity units (NTUs) or Formazin Nephelometric Units (FNUs).

Refer to the appropriate TGI(s) for surface water sampling and/or groundwater sampling, if applicable. In addition, the water quality instruments should be cleaned in accordance the manufacturers specifications and the *TGI for Groundwater and Soil Sampling Equipment Decontamination* and all investigative derived wastes (IDW) should be managed in accordance with project plans.

4 Personnel Qualifications

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, the sampling team will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP), Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Additionally, the field sampling team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

5 Equipment List

The following materials, as required, will be available during field measurement of water qualityparameters:

- Site-specific HASP and health and safety documents identified in the HASP (e.g., job safetyassessments [JSAs])
- Field Implementation Plan (FIP) that includes site map with sampling locations, well constructionrecords (table or logs), sampling plan, and prior sampling records (if available)
- Field notebook, pen(s) (indelible ink) and/or smart device (smart phone or tablet, see Section 10)
- Field laptop / smart device / data manager, as appropriate
- Water Quality Measurement Log (**Attachment 1**) or smart device with a digital form; alternatively, the surface water sampling or groundwater sampling form may be used, if appropriate.
- Water Quality Meter Calibration Log (**Attachment 2**)
- Appropriate personal protective equipment (PPE) (e.g., latex or nitrile gloves, safety glasses, etc.) asspecified in the HASP
- Traffic cones, delineators, and caution tape as appropriate for securing the work area as specified inthe Traffic Safety Plan (TSP)
- Well key(s), as needed
- Photoionization detector (PID), flame ionization detector (FID) or other air monitoring equipment, asneeded, in accordance with the HASP
- Plastic sheeting (e.g., Weatherall Visqueen) or other clean surface to prevent sampling equipment from coming in contact with potentially contaminated surfaces, as needed
- Multiparameter (temperature/pH/specific conductivity/ORP/turbidity/DO) water quality meter or sondewith flow-through cell (as appropriate) plus reader and protective housing, for example:
    - YSI Professional Plus Multiparameter Instrument
    - YSI EX01 or EX02 Multiparameter Sonde
    - Horiba W-22 XD Multiparameter Instrument
    - Hydrolab DS5 Multiprobe and Display

- Turbidity meter (e.g., Lamotte 1974) and sample vessels, as required by FIP or QAPP.
    - Some multiparameter meters have a turbidity sensor; however, the team must verify the sensor will meet the data quality objectives in the FIP or QAPP.
- Operation manual(s) for meter(s)
- Water quality meter extension cable (as needed for in-situ readings of wells)
- Standard solutions for calibration, check expiration dates
- Multimeter maintenance kit and extra DO membranes if using amperometric DO probe
- Extra batteries for the various instruments and/or charging cables
- Electronic water-level indicator (e.g., Solinist Model 101) or oil/water interface probe with 0.01-foot accuracy), as needed
- Tape measure, as needed
- Cleaning equipment (buckets, distilled or deionized water, cleansers appropriate for removingexpected chemicals of concern, paper towels), as needed.

6 Cautions

NOTE: *Proper operation and maintenance are very important for a functioning meter and accuratereadings. Carefully follow manufacturer's instructions for operation (including down-hole use), maintenance, and calibration as each manufacturer's instructions will vary. Some meters require periodic manufacturer calibration.*

Verify the meters meet the project data quality objectives in the FIP and QAPP. In some cases, it may be necessary to use a separate turbidity meter (nephelometer) or higher accuracy and precision meter(s) if the measurements are intended to be used for compliance purposes.

Verify the meter is intended for in-situ measurements (i.e., can be fully submerged in water) and that all necessary parts are available prior tofield mobilization. Check and record the expiration dates of calibration fluids. Verify that calibration readings are reasonableand correspond to previous calibration readings when available. Expired or contaminated calibration fluids may result in erroneous results. If accurate measurements of TDS are required, site-specific calibration will be necessary in accordance with the manufacturer's specifications.

When taking measurements in the field, verify that readings make sense and compare to historicalreadings when possible. See Section 3.

7 Health and Safety Considerations

The HASP will be followed, as appropriate, to ensure the safety of field personnel.

Appropriate personal PPE will be worn at all times in line with the task and the site-specific HASP. Also, at a minimum, wear latex gloves and safety glasses when working with calibration solutions.

Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work.

Access to wells and some surface water bodies may expose field personnel to hazardous materials such as contaminated groundwater ornon-aqueous phase liquid (NAPL) (e.g., oil). Other potential hazards include pressurized wells, stinging insects that may inhabit well heads, other biologic hazards (e.g. ticks in long grass/weeds around wellhead), and potentially the use of sharp cutting tools (scissors, knife)—open well caps slowly and keep face and body away to allow to vent any built-up pressure; only use non-toxic peppermint oil spray for stinging insect nests; review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives, and use appropriate hand protection.

If working near surface water bodies, it may be necessary to use personal flotation devices and/or use additional safety measures. Consult with the HASP and JSA for site- and task-specific health and procedures.

If working at a site with hazardous levels of volatile organic compounds, headspace and/or breathingzone readings will be measured prior to working at/in the well, in accordance with the HASP.

8 Procedures

This Procedures Section is divided into:

- General Procedures – calibration, operation, and maintenance procedures that apply to all methods and meters
- Calibration – general calibration procedures for:
    - Multimeter
    - Separate turbidity meter, if required
- Operation – general measurement operations for:
    - Ex-situ measurements
        - Multimeter
        - Separate turbidity meter, if required
    - In-situ measurements (downhole/well or submerged applications)
- Maintenance
    - Multimeter
    - Turbidity meter

8.1 General Procedures

The following, general procedures apply to most instruments regardless of whether measuring in-situ or ex-situ. For groundwater, most measurement are conducted ex-situ (i.e., not within the groundwater well/aquifer). It is also possible to use in-situ measurements for some parameters and ex-situ measurements for other parameters. If in-situ measurements are carried out, a special probe and/or cables are required.

- Read through all relevant procedures before field implementation and familiarize yourself with the equipment
- Verify all equipment is present and the instrument is fully functioning prior to field mobilization, if possible
    - Check batteries/power levels
- The meter(s) should be cleaned prior to each use in accordance with cleaning and decontamination procedures as specified in the FIP or QAPP and in accordance with the instruction manual.
    - Most meters can only handle light washing with a mild soap and warm water.
    - After cleaning the sensors should be soaked in distilled/deionized water and then rinsed in distilled/deionized water. The recommended soaking periods vary depending on the sensor (see Section 8.2)
    - It is recommended that you temporarily store the probes in distilled/deionized water uses between measurements as some probes like conductivity require long soaking periods (see Section 8.2).
- The meter will be calibrated following the manufacturer's instructions as calibration steps vary bymanufacturer
    - Probes should be rinsed with a small amount of the calibration solution and then discard that used solution prior to calibration, if applicable. This will reduce cross-contamination of the calibration solution during calibration
    - Two or three-point calibrations are preferred over single point calibrations, where applicable. See Section 8.2 and should be selected based on expected water quality
    - Verify calibration solutions, if applicable, are not expired
    - Caution: calibration solutions should not be reused as it can cross-contaminate the solution unless authorized by the QA/QC manager
    - Ensure the sensor is completely submerged in the calibration solution, if applicable
    - Calibration information will be recorded in the calibration log (Attachment 2) and the field notebook
    - More frequent calibration may be necessary in harsh conditions or per project plans
    - Barometric verification or calibration may be required for some instruments
- The meter will be operated following the manufacturer's instructions
- Multimeters can be operated in-situ (down-hole, in water, etc.) or ex-situ (water is transferred to container for measurement)
    - Ex situ operation is described in Section 8.3.1
    - In situ operation is described in Section 8.3.2
- The meter will be maintained according to the manufacturer's instructions
    - Maintenance information will be recorded in the field notebook or instrument log
    - Manufacturer recalibration or replacement probes may be required on a routine basis
    - Additional details for pH, conductivity, temperature, dissolved oxygen, and turbidity maintenance

are presented in section 8.4 Maintenance below

- More frequent maintenance (probe replacement, sensor reconditioning, etc.) may be necessary in harsh conditions or per project plans

- A replacement meter and probes will be available onsite or ready for overnight shipment, as necessary
- Store the meters in accordance with manufacturer's specifications
    - Most multi-meter sensors should be stored with a small amount of water in the cap to keep it moist but not saturated/submerged.

The complete procedures are described in Section 8.2 through 8.4.

8.2 Calibration

8.2.1 Multimeter Calibration

1) Connect cables from meter to reader
2) Check probes and ensure they are clean
3) Switch on instrument and allow to warm-up, warm up may take from 5 to 15 minutes depending on the instrument and sensors used
4) Check battery life and replace, if needed
5) Adjust date/time, if needed
6) Soak all probes in distilled or de-ionized water for at least 5 minutes (some probes may requirelonger soak times, see below and manufacturer instructions) and then shake off excess liquid
    i. NOTE: *If the probes are recently reconditioned or have slight build-up, allow for longer soaking period*
7) Navigate to calibration display/mode; enable auto stabilization feature, if appropriate
8) **pH Calibration** – when in use, the pH meter will be calibrated daily, at a minimum.
    a. Connect electrode (if applicable) or remove protective cap from electrode
    b. Rinse end of electrode in distilled/deionized water and shake off excess water
    c. Measure and record temperature of buffer solutions
    d. Immerse pH electrode in pH buffer 7.00, set the temperature to that of the buffer 7.00, andallow sufficient time for the electrode to stabilize
    e. Adjust the calibration for the correct readout and temperature
    f. Confirm/press the calibration button
    g. Remove electrode from buffer and rinse with distilled/deionized water
    h. Immerse pH electrode in buffer 4.00, set the temperature control to that of the buffer 4.00,and allow sufficient time for the electrode to stabilize

i. Adjust the calibration for the correct readout and temperature

j. Confirm/press the calibration button

k. Rinse electrode with distilled/deionized water

l. A pH 10 calibration will also be performed if high pH (>9) is anticipated, following manufacturers procedures

m. The pH meter is calibrated

9) **Conductivity Calibration** – when in use, the conductivity meter will be calibrated daily, at aminimum.

a. Conductivity is generally measured in specific conductivity (temperature compensated),verify with the FIP/work plan

b. Soak the probe in distilled/deionized water for at least 30 minutes

c. Remove the probe from the water and fling out drops clinging inside

d. Immerse the probe to or beyond the vent holes in a beaker containing 1.413 mS/cmstandard solution for freshwater measurements or a standard with a higher concentration for marine water, and gently agitate vertically to remove entrapped air

e. Repeat Steps 3 and 4 at least once more

f. Press calibration button

10) **Temperature calibration** – temperature will be verified according to FIP/work plan, if applicable.

11) **Dissolved Oxygen (DO) calibration** – when in use, the DO meter will be calibrated daily usingthe air calibration method or less frequently if using an optical sensor (see below).

a. Preparation

i. Polarographic sensor – periodically recondition sensor, replace fluids, and Teflon®membrane, per manufacturer, and air bubbles should not be present

ii. Galvanic sensor – Periodically recondition sensor, replace fluids, and membrane,per manufacturer, and air bubbles should not be present

iii. Optical sensor – Per manufacturer, most will only require field checks and maintenance, however, weekly checks are recommended to verify the accuracy, ata minimum

b. Obtain a barometric pressure reading from a daily weather report or from the instrument,as required by instrument

c. Keep instrument upright and vent cap/cover while retaining a small amount of clean water(do not cover probes) for non-optical sensors only

d. Allow 5 to 15 minutes for optimum probe stabilization and polarization, for non-optical sensors (reading will range between 5 to 9 mg/L depending on temperature and barometricpressure)

e. Press calibration button if reading is more than 2% from the standard

12) **Oxidation reduction potential (ORP) calibration** – when in use, the ORP meter will becalibrated daily, if required.
    a. Rinse the probe in distilled/deionized water
    b. Remove the probe from the water and fling out drops clinging inside
    c. Immerse the probe in the ORP solution. Gently agitate vertically to remove entrapped air.
    d. Adjust calibration target based on temperature in accordance with manufacturer and calibration solution guide
    e. Allow the sensor to equilibrate to solution
    f. Press calibration button if reading is more than 2% from the standard
13) **Turbidity, if used** – when in use, turbidity sensor will be checked daily and calibrated weekly, at aminimum
    a. Verify with the FIP/work plan if the turbidity sensor will be used and the proper units (NTU, FNU, etc.)
    b. Gently clean the sensor to remove and sediments or buildup and then rinse the sensor with distilled/deionized water one to two times
    c. Remove the probe from the water and fling out drops clinging inside
    d. Next do a final rinse with the calibration solution starting with lower calibration solution and dispose of the used solution.
    e. Immerse the probe in the calibration cup with the guard. It is important to calibrate with the guard on or verify the cup is designed to match the guard as turbidity measurements are affected by the dimensions and reflectivity of the guard. Verify the there are no air bubbles.
    f. Repeat Steps c, d, and e at least once more with higher turbidity calibration solutions
    g. Press calibration button
14) Adjust the instrument to READ or LOGGING mode, per instruction manual and project plans; theinstrument is now ready for use
15) Record the calibration on the field form or smart device.

8.2.2 Turbidity Meter Calibration (if required)

When in use, the turbidity meter will be checked daily and calibrated weekly. The turbidity meter will be calibrated per steps below:

1) Switch on instrument and allow time to warm-up
2) Check battery life and replace, if needed
3) Turbidity sample tubes will always be washed prior to use, but not the calibration standard tubes,using a mild detergent to remove any dirt or fingerprints that could bias the results
4) Dry the outside of the sample turbidity tubes with a clean, lint-free cloth or disposable wipe (non-scratch)

andallow the turbidity tubes to air-dry in an inverted position to prevent dust from entering the tube,as allowable (but, not the calibration standard tubes)—dirt or fingerprints can lead to inaccurateresults.

5) Wipe the 1 NTU standard and place in the chamber after aligning the arrows (manufacturer dependent)

6) Place the light shield over the turbidity standard and allow the meter to stabilize

7) Press the read and/or CAL button, per instructions

8) Repeat steps 3 thru 5 above

9) Adjust to READ mode and the instrument is now ready for use

10) Record the calibration and/or verification in the calibration log.

8.3 Operation

Water quality parameters can be measured ex-situ or in-situ in accordance with the FIP/work plan. The operating procedures for each method are described below. It is also possible to collect a depth-specific water sample from a well or water body for ex-situ measurement.

8.3.1 Ex-situ Operation

For ex-situ operation one or two representative water samples will be collected from the well or surface water body for measurement using the required meters. If two representative samples are collected, both values for each parameter should be reported, however, the average of the two results should be used as the final value.

8.3.1.1 Multimeter

1) Fill two 100-mL plastic clean, disposable containers or cleaned beakers with water from the sample

2) Insert the probe into the first beaker immersing all sensors and allow to stabilize (2 minutes atminimum)

3) Record readings

   a. Temperature – in degrees Celsius or Fahrenheit

   b. pH – in standard units between 0 and 14, in 0.01 increments

   c. Specific conductivity – in mS/cm or micro siemens per centimeter (uS/cm)

   d. DO – in mg/L and percent (will typically read between 0 and 15 mg/L)

   e. ORP – in millivolts (mV; will typically read between -1,400 mV and + 1,400 mV), if required

   f. Turbidity, if used – in NTU or NFU

4) Rinse probe off with distilled/deionized water

5) Repeat Steps 2 thru 4 for the other beaker, if applicable

6) Record results on the water quality measurement log and/or in field notebook - the average will bethe actual result, after stabilization if two samples are used

7) Rinse probe off with distilled/deionized water

NOTE: *If a flow through cell is used during sampling, a single reading can be obtained from the multimeter during each step of the wells purging in accordance with TGI - Low FlowGroundwater Purging and Sampling.*

8.3.1.2 Turbidity Meter, if needed

1) Fill the two cleaned, manufacturer provided sample tubes with water from the sample
    a. If using a flow through cell, it is recommended to collect the turbidity samples before the flow through cell
2) Wipe off and dry the outside of the sample turbidity tubes with a clean, lint-free cloth ordisposable wipe
3) Insert the first sample tube and close the cover
4) Push the READ button
5) Record turbidity reading in NTUs (0 to 1,100 NTUs)
    a. If readings are over the instrument limit, a single 2X dilution can be run by mixing thesample with equal parts distilled or de-ionized water and gently mixing
    b. The resulting value will need to be doubled (for example, if the diluted reading is 750 NTUs, the estimated turbidity would be 750 x 2 = 1,500 NTUs. Diluted samples will bequalified as estimated)
6) Repeat steps 2 thru 5 with the second sample tube
7) Log the results on the water quality measurement log and/or in field notebook - the average willbe the actual result
8) Rinse sample tubes with distilled/deionized water.

NOTE: *If a flow through cell is used during low flow groundwater sampling, a single reading can be obtained from the multimeter during each step of the wells purging in accordance with TGI - Low FlowGroundwater Purging and Sampling. The turbidity sample is recommended to be collected prior to passing through the flow through cell.*

8.3.2 In-situ Operation

1) Connect extension cable and protective housing to meter
2) Measure water level from reference point
3) Lay out plastic sheeting as needed to keep the multimeter clean
4) Clean multimeter and cable per decontamination procedures and instruction manual and rinse with distilled/deionized water using caution, as most meters can only handle light washing with amild soap and warm water
5) Organize work area to prevent dirt or objects from falling in the well
6) Measure and mark extension cable at planned measurement intervals in accordance with the FIP
7) Slowly lower the probe into the well or water body to the desired measurement interval usingcaution to prevent rubbing of cable on the well and minimize water and sediment disturbance

8) Allow readings to stabilize (typically 1 to 3 minutes)
    a. For many instruments, the instrument may need to be slowly oscillated up and down a fewinches to circulate water around the probes
9) Record readings
    a. Temperature – in degrees Celsius or Fahrenheit
    b. pH – in standard units between 0 and 14, in 0.01 increments
    c. Specific conductivity – in mS/cm or uS/cm
    d. DO – in mg/L and percent (will typically read between 0 and 15 mg/L)
    e. ORP – in mV (will typically read between -1,400 mV and +1,400 mV), if required
    f. Turbidity, if used – in NTU or NFU
10) Repeat steps 4 through 9 to complete targeted in-situ measurements
11) Record results on the water quality measurement log or smart device
12) Slowly retrieve the multimeter
13) Clean multimeter and cable per decontamination procedures and instruction manual and rinse with distilled/deionized water using caution, as most meters can only handle light washing with amild soap and warm water

8.4 Maintenance

8.4.1 Multimeter

1) After use, the meter will be inspected and maintained according to the manufacturer's specifications and the inspection/maintenance activities will be recorded in the field notebook
2) Keep records of usage, maintenance, calibration, problems, and repairs
3) Recharge/replace batteries on a regular basis
4) Store meters or electrodes in protective casing when not in use
5) DO membranes will be stored moist, unless specified otherwise by manufacturer
6) Focused probe cleaning may be necessary in accordance with the operation manual and may be needed more frequently in harsh conditions
7) A replacement meter will be available onsite or ready for rapid delivery/shipment
8) Periodic manufacturer calibration may be necessary per operation manual and/or field plans.

8.4.2 Turbidity Meter, if used

1) Recharge battery on a regular basis

2) Store in protective casing when not in use

3) Keep records of usage, maintenance, calibration, problems, and repairs

4) After use, the meter will be inspected with results recorded in the field notebook

5) Keep sample tubes clean inside and out replacing them when they become scratched or etchedand avoid handling the tubes in the region where the light beam enters them.

6) Clean lens periodically per instruction manual

7) Turbidity meter will be sent back to the manufacturer for service when needed

9 Waste Management

IDW, including decontamination liquids, and disposable materials (well material packages, PPE, etc.), will be placed in clearly labeled, appropriate containers, or managed as otherwise specified in the Work Plan (or equivalent), FSP, and/or IDW management guidance document.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The FieldNow® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Management of the original documents from the field will be completed in accordance with the site- specific QAPP. Records generated as a result of this TGI will be controlled and maintained in the projectrecord files in accordance with project requirements.

In general, documentation of the following information is required:

- Calibration – calibration information will be recorded on a calibration form, field log, or electronicdevice per project plans
    - Meter manufacturer and model
    - Serial number
    - Calibration personnel
    - Calibration date/time
    - Standard value, initial and final reading
    - Observations, if applicable

- Readings - data will be recorded on a field log, sampling form, or electronic device per projectplans
    - Instrument model
    - Measurement date/time
    - Field personnel
    - Weather
    - Measurement location and depth, if applicable
    - Source of water (surface water, groundwater, etc.)
    - In-situ vs ex-situ measurement method
    - Value of readings and average reading, if applicable
    - Units of readings
    - Key observations

All records will be provided to the project manager and retained in the project files. Any maintenanceneeds will be communicated to the project manager promptly.

11 Quality Assurance

Quality assurance procedures will be conducted in accordance with the Arcadis Quality ManagementSystem or the site-specific QAPP. Refer to the QAPP or FIP/sampling plan/work plan for the frequency of calibrations.

12 References

Not applicable.

13 Attachments

Attachment A - Water Quality Measurement Log

Attachment B - Water Quality Meter Calibration Log

Attachment A

Water Quality Measurement Log

WATER QUALITY MEASUREMENT LOG

GENERAL INFORMATION

Date _____ Weather _____

Project No _____ Sample Personnel _____

Site Name _____ Sample ID _____

Site Location _____ Start reading time _____

Site/Well No. _____ End reading time _____

MEASUREMENT DATA

Measuring Point (MP) description _____ ☐ Surface Water or ☐ Groundwater

Depth to Water (ft)/Time _____ Instrument model/serial no. _____

Well Screen Interval (ft) _____ Instrument model /serial no. 2 _____

Casing Diameter (in) _____ ☐ In-situ ☐ Ex-situ reading

Meters calibrated (Y/N) _____ (see calibration log, if applicable)

Water Quality Parameters

Time	Depth (ft)	pH (S.U.)	Spec. Cond. (mS/cm or uS/cm)	Temp (°C or °F)	DO (mg/L)	DO (%)	ORP (mV)	Turbidity (NTU)	Observations (Odor, clarity, etc., if applicable)

REMARKS

_____

_____

_____

_____

Attachment B

Water Quality Meter Calibration Log

WATER QUALITY METER CALIBRATION LOG

PROJECT NAME	
PROJECT NO.	
MODEL(s)	
SERIAL #(s)	
SAMPLER	
DATE	

TURBIDITY CALIBRATION

CAL. READING		CAL. READING		CAL. RANGE	TIME
(LOT #)		(LOT #)			
(EXP. DATE)		(EXP. DATE)			
PRE-CAL. / POST-CAL		PRE-CAL. / POST-CAL			
/		/		☐ WITHIN RANGE	
/		/		☐ WITHIN RANGE	
/		/		☐ WITHIN RANGE	
/		/		☐ WITHIN RANGE	

MULTIMETER CALIBRATION

AUTOCAL SOLUTION (Y / N)			CAL. READING	CAL. RANGE	TIME	CALIBRATION RANGES [1]	
(LOT #)							
(EXP. DATE)							
PARAMETER		LOT / EXP. DATE	PRE-CAL. / POST-CAL				
☐	pH		/	☐ WITHIN RANGE		pH	± 0.2 S.U.
☐	CONDUCTIVITY		/	☐ WITHIN RANGE		COND	± 1% OF CAL. STANDARD
☐	ORP		/	☐ WITHIN RANGE		ORP	± 25 mV
☐	DO		/	☐ WITHIN RANGE		DO	Atmospheric
☐	TURBIDITY		/	☐ WITHIN RANGE		TURB	± 5% OF CAL. STANDARD
☐			/	☐ WITHIN RANGE			
☐			/	☐ WITHIN RANGE			

(1) CALIBRATION RANGES ARE SPECIFIC TO THE MODEL OF THE WATER QUALITY METER

NOTES:

SIGNED DATE

CHECKED BY DATE

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TGI – Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells

Rev: 3

Rev Date: April 5, 2023

Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
	0	October 12, 2018	All	Updated and re-written as TGI with new branding and content	Marc Killingstad
	1	May 8, 2020	Pages 5, 10-11	Added clarification/details for equipment requirements and procedure steps based on USEPA guidance	Marc Killingstad
	2	April 5, 2022	All	Updated to new branding template and minor edits	Marc Killingstad
	3	April 5, 2023	All	Annual review completed by SME. Document version number and document date updated.	Marc Killingstad

Approval Signatures

Prepared by:	Xuan Xu	4/5/2023
	Xuan Xu (Preparer)	Date
Reviewed by:		4/5/2023
	Marc Killingstad (Subject Matter Expert)	Date

1 Introduction

Groundwater samples are collected from monitoring wells to evaluate groundwater quality. The protocol presented in this Technical Guidance Instruction (TGI) describes the procedures to purge monitoring wells and collect groundwater samples using the low flow purging/sampling methodology. This protocol has been developed in accordance with the United States Environmental Protection Agency (USEPA) Region I *Low Stress (Low Flow) Purging and Sampling Procedures for the Collection of Groundwater Samples from Monitoring Wells* (EQASOP-GW4; September 19, 2017).

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

Both filtered and unfiltered groundwater samples may be collected using this low-flow sampling method. Filtered samples will be obtained using a 0.45-micron disposable filter. Project teams will evaluate the last time the monitoring wells were developed and determine if additional development might be necessary. Water samples will not be taken immediately following well development. Sufficient time will be allowed for the groundwater flow regime in the vicinity of the monitoring well to stabilize and to approach chemical equilibrium with the well

construction materials. This lag time will depend on site conditions and methods of installation but often exceeds one week.

4 Personnel Qualifications

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations,such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, the groundwater sampling team will review and be thoroughly familiar withrelevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP)/field sampling plan, Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Additionally, the groundwater sampling team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

5 Equipment List

Specific to this activity, the following materials (or equivalent) will be used:

- Site-specific HASP and health and safety documents identified in the HASP
- Field Implementation Plan (FIP) that includes site map, well construction records, sampling plan (sample analyses, sample volume required, and sample holding time), and prior groundwater sampling records (if available)
- Field notebook and/or smart device (phone or tablet)
- Low-flow sampling field forms (**Attachment A**)
- Appropriate personal protective equipment (PPE) (e.g., latex or nitrile gloves, safety glasses, etc.)as specified in the HASP
- Well keys and other tools to remove manhole covers (manual torque wrench with 9/16” socket and flat head screwdriver typical)
- Photoionization detector (PID) or Flame ionization detector (FID) (as appropriate, depending onsite-specific constituents of concern)
- Electronic water-level indicator (e.g., Solinist Model 101) or oil/water interface probe with 0.01- foot accuracy (oil/water as appropriate, note that sampling will not be performed when sheen orlight non-aqueous phase liquid [LNAPL] is present)
- Down-hole multi-parameter water-quality sonde (temperature/pH/specific conductivity/oxidationreduction [ORP]/turbidity/dissolved oxygen) meter coupled with flow-through-cell for measurements, for example:

  - YSI 6-Series Multi-Parameter Instrument
  - Horiba U-22 Multi-Parameter Instrument.
  - Hydrolab Series 3 or Series 4a Multiprobe and Display.

  NOTE: *Transparent, small volume flow-through-cells (e.g., 250 milliliters or less) are preferred as they allow for easy detection of air bubbles and sediment buildup in the cell, which can interfere with the monitoring instrument probes. A small volume cell also allows for quick turnover of water in the cell between measurements of the indicator field parameters. It is recommended to use a flow-through-cell and monitoring probes from the same manufacturer and model to avoid incompatibility between the probes and flow-through-cell.*

- Plastic sheeting (e.g., Weatherall Visqueen) to protect all down-hole sampling equipment from contact with potential sources of contamination.
- Decontamination equipment
  - Non-phosphate laboratory soap (Alconox or equivalent), brushes, and clean buckets, and/or clean wash tubs—new buckets or tubs will be purchased if it cannot be determined if the present items are clean
  - Distilled or de-ionized water for equipment decontamination
- Indelible ink pen
- 150-foot measuring tape (or sufficient length for the maximum site depth requirement)
- Sampling pump, which may consist of one or more of the following:
  - Submersible pump (e.g., Grundfos Redi-Flo 2)
  - Peristaltic pump (e.g., ISCO Model 150)
  - Bladder pump (e.g., Marschalk System 1, QED Micropurge, Geotech)
- Appropriate controller and power source for pump:
  - Submersible and peristaltic pumps require electric power from either a generator or a deep cell battery
  - Submersible pumps such as Grundfos require a pump controller to run the pump
  - Bladder pumps require a pump controller and a gas source (e.g., air compressor or compressed N_2 or CO_2 gas cylinders)
- Teflon® tubing or Teflon®-lined polyethylene tubing of an appropriate size for the pump being used
  - For peristaltic pumps, dedicated Tygon® tubing (or other type as specified by the manufacturer) will be used through the pump apparatus
  - Teflon® will not be used when sampling for per- and polyfluoroalkyl substances (PFAS)
- Graduated cylinder and stopwatch or other device to measure time to determine pumping rate
- Appropriate water sample containers (supplied by the laboratory)
- Appropriate blanks (trip blank supplied by the laboratory)
- Sample labels and Chain-of-Custody forms (COC)
- 0.45-micron disposable filters (if field filtering is required)

- A supplemental turbidity meter (e.g., Horiba U-10, Hach 2100P, LaMotte 2020) may be required for specific projects and will be specified in the project FIP/ work plan and the kick-off notes.
    - If used, in-line 'T' and valve allows for collection of water for turbidity measurements before the pump discharge enters the flow-through cell

NOTE: *The maintenance requirements for the above equipment generally involve decontamination or periodic cleaning, battery charging, and proper storage, as specified by the manufacturer. For operational difficulties, the equipment will be serviced by a qualified technician.*

6 Cautions

Different USEPA regions and/or state regulatory agencies may stipulate deviations from this document. Itis the responsibility of the Project Team (Project Manager and Technical Lead) to be fully aware of the requirements from the applicable regulatory framework.

Weather

- If heavy precipitation occurs, and no cover over the sampling area and monitoring well can be erected, sampling may be discontinued until adequate cover is provided. Rainwater could compromise groundwater samples.
- Avoid extreme weather situations. Be aware that thermal currents and vertical mixing of cold and warm water inside the well casing could create a convection cell within the well and compromise data collection (e.g., biological mechanisms).
    - Direct sunlight and hot ambient temperatures may cause the groundwater in the tubing orflow-through-cell to heat up and de-gas. This may result in the loss of volatile organic compounds (VOCs) and dissolved gases. Shade the equipment from direct sunlight, keep the tubing as short as possible and avoid the hottest times of the day.
    - Sampling during freezing conditions may adversely impact the data quality objectives. USEPA recommends low-flow sampling be conducted at air temperatures above 32°F (0°C) or taking special precautions to prevent groundwater from freezing in the equipment.

Cross-Contamination

- To mitigate potential cross-contamination, groundwater samples are to be collected in a pre- determined order from least impacted to impacted based on previous analytical data. If no analytical data are available, collect samples in order of up-gradient, then furthest down-gradient tosource area locations.
- Note that permanent markers could introduce volatile constituents into the samples*; therefore, indelible ink is recommended* to be used for labels on sample containers or sample coolers.
- When using a gasoline generator, this power source will be set-up at least 30 feet downwind from the well to avoid exhaust fumes to contaminate samples.

Pumps

- Preferred methods of extracting groundwater are adjustable rate, submersible pumps - such as centrifugal pumps or bladder pumps – constructed of stainless steel or polytetrafluoroethylene (PTFE, i.e., Teflon®). However, *PTFE will not be used when sampling for per- and polyfluoroalkyl substances (PFAS). PTFE could contain PFAS*.

- When using a bladder pump for collecting VOCs and dissolved gases, “best practice” is to set-up the pump to deliver sufficient water to fill a 40 mL VOC vial.
- The use of peristaltic pumps will be based on the type of data to be collected. *Because the use a peristaltic pump can result in de-gassing of VOC and / or dissolved gases from groundwater, a different type of pump will be considered if these compounds are of concern.*
- *Manual or motor driven inertial pumping devices are not recommended because they cause greater disturbance during purging and pumping than regular pumps and are less easily controlled. This could cause a higher degree of data variability.*

Tubing

- When sampling for VOCs, SVOCs, pesticides, PCBs and inorganics, use of PTFE (Teflon®) or PTFE-lined tubing is preferred. However, PTFE tubing will not be used when sampling for PFAS.
- PVC, polypropylene or polyethelene tubing may be used when sampling for metals or other inorganics.
- Tubing with inside diameters of 1/4 or 3/8 inch is recommended because this will help ensure tubing remains water filled when operating at very low pumping rates.

General Precautions

- Store and/or stage empty and full sample containers and coolers out of direct sunlight.
- It may be necessary to field filter the groundwater for some parameters (e.g., metals) during collection, depending on preservation, analytical method, and project quality objectives. The task-kick-off notes and the FIP/work plan will list the samples that require field filtering.
- Be careful not to overtighten lids with Teflon® liners or septa (e.g., 40 mL vials). Over-tightening can cause the glass to shatter or impair the integrity of the Teflon® seal.

7 Health and Safety Considerations

The HASP will be followed, as appropriate, to ensure the safety of field personnel.

Appropriate personal protective equipment (PPE) will be always worn in line with the task and the site-specific HASP.

Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work.

Access to wells may expose field personnel to hazardous materials such as contaminated groundwater or non-aqueous phase liquid (NAPL) (e.g., oil). Other potential hazards include pressurized wells, stinging insects that may inhabit well heads, other biologic hazards (e.g. ticks in long grass/weeds around well head), and potentially the use of sharp cutting tools (scissors, knife)—open well caps slowly and keep face and body away to allow to vent any built-up pressure; only use non-toxic peppermint oil spray for stinging insect nests; review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives, and use appropriate hand protection.

Generators and cord and plug equipment will employ an overcurrent protection device such as an integrated ground fault circuit interrupter (GFCI) cord. Grundfos pump controllers will not run properly with a GFCI, so the power source will be equipped with other overcurrent protection means.

Overtightening of lids with Teflon® liners can cause the glass to shatter and create a risk for hand injuries.

8 Procedure

Field personnel will set up and perform low-flow sampling in accordance with the following procedures.

1. Review FIP and groundwater sampling records from previous sampling events (if available) prior to mobilization to estimate the optimum pumping rate and anticipated drawdown for each well to perform sampling as efficiently as possible (i.e., reach a stabilized pumping condition).
2. Calibrate field instruments according to manufacturer procedures for calibration and record calibration procedure and results in field log.
3. All equipment will either be new or decontaminated in accordance with appropriate guidance document *(TGI – Groundwater and Soil Sampling Equipment Decontamination)* prior to use.
4. Visually inspect the well to ensure that it is undamaged, properly labeled and secured
   a. Damage or other conditions that may affect the integrity of the well will be recorded in the Field Activity Daily Log and brought to the attention of the designated Field Manager and/or Project Manager
   b. Record well construction and conditions on the Low-Flow Sampling Field Form **(Attachment A)**.
5. Place clean plastic sheeting on the ground near the well to keep monitoring and sampling equipment off the surface unless the equipment is elevated above the ground (e.g., on a table).
6. Open the well cover while standing upwind of the well. Remove the well cap and place it on the plastic sheeting. If appropriate or required for site-specific conditions, insert the photoionization detector (PID) probe approximately 4 to 6 inches into the casing or the well headspace and cover it with a gloved hand. Record the PID reading in the field log. Perform air monitoring in the breathing zone according to the HASP and/or JSA.
7. Measure and record the initial depth to groundwater prior to placing the pumps.
8. Prepare and install the pump in the well.

   NOTE: *Groundwater will be purged from the wells using an appropriate pump. If the depth to water is below the sampling range of a peristaltic pump (approximately 25 feet below ground surface), a submersible or bladder pump will be used, provided that the well is constructed with a casing diameter of at least two (2) inches (the minimum well diameter capable of accommodating such pumps). For smaller diameter wells, where the depth to water is below the sampling range of a peristaltic pump, alternative sampling methods (i.e., bailing or small diameter bladder pumps) will be used to purge and sample the groundwater. Bladder pumps are preferred over peristaltic and submersible pumps to prevent volatilization if sampling of VOCs and/or dissolved gasses is required. Purge water will be collected and containerized according to the direction of the project team.*

   a. For submersible and non-dedicated bladder pumps, decontaminate the pump according to site decontamination procedures. Non-dedicated bladder pumps will require a new bladder and attachment of an air-line, sample discharge line, and safety cable prior to placement in the well. Attach the air-line tubing to the air-port on the top of the bladder pump. Attach the sample discharge tubing to the water port on the top of the bladder pump. Take care not to reverse the air and discharge tubing lines during bladder pump setup, as this could result in bladder failure or rupture. Attach and secure a safety cable to the eyebolt on the top of pump (if present, depending on pump model used). Slowly lower the pump, safety cable, tubing, and electrical lines into the well to a depth corresponding to the approximate center of the saturated screen section of the well. Avoid twisting

and tangling of safety cable, tubing, and electrical lines while lowering the pump into the well; twisted and tangled lines could result in the pump becoming stuck in the well casing. Also, make sure to keep tubing and lines from touching the ground or other surfaces while introducing them into the well, as this could lead to unintended contamination.

b. If using a bladder pump, connect the air-line to the pump controller output port. The pump controller will be connected to a supply line from an air compressor or compressed gas cylinder using an appropriate regulator and air hose. Tighten the regulator connector onto the gas cylinder (if used) to prevent leaks. Teflon® tape may be used on the threads of the cylinder to provide a tighter seal. Once the air compressor or gas cylinder is connected to the pump controller, turn on the compressor or open the valve on the cylinder to begin the gas flow. Turn on the pump controller power (if an on/off switch is present) and verify that all batteries are charged and fully functioning before starting the pump.

c. If a peristaltic pump is being used, slowly lower the sampling tubing into the well to a depth corresponding to the approximate center of the saturated screen section of the well. The pump intake or sampling tube must be kept at least two (2) feet above the bottom of the well to prevent mobilization of any sediment present in the bottom of the well.

d. If using an in-line 'T' and valve, install between pump discharge water line and the bottom inlet port of the flow-through cell. Attach a short piece of tubing to the outlet. This set-up will be used to collect samples for turbidity readings.

9. Connect the pump discharge water line to the bottom inlet port on the flow-through cell connected to the multi-parameter water-quality sonde and make sure to record equipment/instrument identification (manufacturer and model number).

10. Before starting the pump, ensure that the water level inside the well has stabilized (i.e., measure the water level multiple times after deploying the pump in the well).

11. Start pumping the well at 200 to 500 milliliters (mL) per minute (or at lower site-specific rate if specified) and adjust the pumping rate to cause little or no water level drawdown in the well (less than 0.3 feet below the initial static depth to water measurement): the water level should stabilize, however, this is not always possible.

12. If the well diameter is of sufficient size, measure the water level every 3 to 5 minutes (or as appropriate, lower flow rates may require longer time between readings) during pumping.

13. Maintain a steady flow rate to the extent practicable and do not break pump suction or cause entrainment of air in the sample.

14. Record pumping rate adjustments and depths to water.

    If necessary, reduce pumping rates to the minimum capabilities of the pump to avoid pumping the well dry and/or to stabilize indicator parameters; if the recharge rate of the well is very low, use alternative purging techniques, which will vary based on the well construction and screen position.

    For wells screened across the water table, the well may be pumped dry, and sampling can commence as soon as the volume in the well has recovered sufficiently to permit collection of samples.

    For wells screened entirely below the water table, the well can be pumped until a stabilized level (which may be greater than the maximum displacement goal of 0.3 feet) is maintained and monitoring for stabilization of field indicator parameters can commence; if a lower stabilization level cannot be

maintained, the well may be pumped until the drawdown is at a level slightly higher than top of the well screen.

15. After water levels have stabilized and a sufficient volume has been purged (see note below), continue pumping and begin monitoring field indicator parameters using a multi-parameter water-quality sonde coupled with a flow-through-cell.

    NOTE: *The final purge volume must be greater than the stabilized drawdown volume plus the pump's tubing volume. If the drawdown has exceeded 0.3 feet and stabilizes, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are collected.*

16. Use the flow to measure all indicator field parameters, except for turbidity, every 3 to 5 minutes (or after each volume of the flow-through cell has been purged or other appropriate interval); turbidity samples will be collected before the flow-through-cell using the T-valve and a clean container such as a glass beaker.

17. Record field indicator parameters on the groundwater sampling log.

18. The well is considered stabilized and ready for sample collection when three consecutive readings are within the following limits:

    - **Turbidity** within ± 10% for values greater than 5 nephelometric turbidity units [NTUs] or if three turbidity values are less than 5 NTUs, consider the values stabilized
    - **Dissolved Oxygen (DO)** within ± 10% for values greater than 0.5 mg/L or if three DO values are less than 0.5 mg/L, consider the values stabilized
    - **Specific Conductance** within ± 3%
    - **Temperature** within ± 3%
    - **pH** within ± 0.1 unit
    - **Oxidation/Reduction Potential (ORP)** within ±10 millivolts (mV)

    NOTE: *Alternate stabilization goals may exist in different geographic regions, consult the site- specific FIP/work plan for stabilization criteria).*

    NOTE: *While achieving turbidity levels less than 5 NTU and a stable drawdown of less than 0.3 feet is desirable, sample collection may still take place provided the indicator field parameter criteria in this procedure are met.*

19. If the parameters have stabilized but turbidity remains relatively high (e.g., greater than 50 NTUs), the pump flow rate may be decreased to a minimum rate of 100 mL/min to reduce turbidity levels as low as possible. If groundwater turbidity has been minimized (i.e., consecutive readings within ± 10%) and the values for all other parameters have stabilized, the well may be sampled; however, consult specifications in the FIP/work plan and/or the project technical lead prior to sampling.

20. If after one (1) hour of purging indicator field parameters have not stabilized, consult specifications in the FIP/work plan and/or the project technical lead prior to sampling.

    In general, three potential options are available if stabilization criteria are not met:

    a. Continue purging until stabilization is achieved.

    b. Discontinue purging, do not collect any samples, and record in field logbook/on the sampling form that stabilization could not be achieved (documentation must describe attempts to achieve stabilization).

c. Discontinue purging, collect samples, and provide full explanation of attempts to achieve stabilization. There is a risk that the analytical data obtained under these conditions, particularly metals and hydrophobic organic analytes, may reflect a sampling bias and, as a result, the data may not meet the data quality objectives of the sampling event.

NOTE: *DO is extremely susceptible to various external influences (including temperature or the presence of bubbles on the DO meter); therefore, great care will be taken to minimize the agitation or other disturbance of water within the flow-through cell while collecting these measurements. If air bubbles are present on the DO probe or in the discharge tubing, remove them before taking a measurement. If DO values are not within acceptable range for the temperature of groundwater, again check for and remove air bubbles on the probe before re-measuring. The table below may be used as a general guide for DO values under various temperatures; however, understand that the table corresponds to freshwater solubility and groundwater contaminants may affect oxygen solubility. If DO value is 0.00 or less, then the meter will be serviced and re-calibrated. If DO values are above possible results, then the meter will be serviced and re-calibrated.*

NOTE: *During extreme weather conditions, stabilization of field indicator parameters may be difficult to attain. Modifications to the sampling procedures to alleviate these conditions (e.g., measuring the water temperature in the well adjacent to the pump intake) will be documented in the field logbook/on the sampling form.*

NOTE: *If other field conditions are suspected of preventing stabilization of certain parameters, detailed observations will be documented in the field logbook/on the sampling form.*

Oxygen Solubility in Fresh Water

Temperature (degrees C)	Dissolved Oxygen (mg/L)
0	14.6
1	14.19
2	13.81
3	13.44
4	13.09
5	12.75
6	12.43
7	12.12
8	11.83
9	11.55
10	11.27
11	11.01
12	10.76
13	10.52
14	10.29
15	10.07
16	9.85
17	9.65
18	9.45
19	9.26
20	9.07
21	8.9
22	8.72
23	8.56
24	8.4
25	8.24
26	8.09
27	7.95
28	7.81
29	7.67
30	7.54
31	7.41
32	7.28
33	7.16
34	7.05
35	6.93

Reference: Vesilind, P.A., Introduction to Environmental Engineering, PWS Publishing Company, Boston, 468 pages (1996)

21. Complete the sample label(s) and cover the label(s) with clear packing tape to secure the label onto the container.
22. After the indicator parameters have stabilized, collect groundwater samples by diverting flow out of the unfiltered discharge tubing into the appropriate labeled sample container.
    a. If a flow-through analytical cell is being used to measure field parameters, the flow-through cell will be disconnected after stabilization of the field indicator parameters and prior to groundwater sample collection.
    b. Under no circumstances will analytical samples be collected from the discharge of the flow- through cell.
    c. If an in-line ‘T’ and valve are used, the valve needs to be removed as well.

d. Samples will be collected in the following order: VOCs, total organic carbon (TOC), semi- volatile organic compounds (SVOCs), metals and cyanide, and others (or other order as defined in the site-specific FIP/work plan).
e. When the container is full, tightly screw on the cap.

23. If sampling for total and filtered metals and/or polychlorinated biphenyls (PCBs), a filtered and unfiltered sample will be collected.
    a. Install an in-line, disposable 0.45-micron particle filter on the discharge tubing after the appropriate unfiltered groundwater sample has been collected.
    b. Continue to run the pump until an initial volume of "flush" water has been run through the filter in accordance with the manufacturer's directions (generally 100 to 300 mL).
    c. Collect the filtered groundwater sample by diverting flow out of the filter into the appropriately labeled sample container.
    d. When the container is full, tightly screw on the cap.
24. Secure with packing material and store the samples on ice in an insulated transport container provided by the laboratory and include a temperature blank in each container to be shipped.
25. Record on the Low-Flow Sampling Field Form (and bound field logbook) the time at which sampling procedures were completed, any pertinent observations of the sample (e.g., physical appearance and the presence or lack of odors or sheens), and the values of the stabilized field indicator parameters as measured during the final reading during purging **(see Attachment A)**.
26. Turn off the pump and air compressor or close the gas cylinder valve if using a bladder pump setup.
27. Slowly remove the pump, tubing, lines, and safety cable from the well.
    a. If using dedicated tubing, do not allow the tubing or lines to touch the ground or any other surfaces which could contaminate them.
    b. If using dedicated tubing, it will be folded - without pinching it - to a length that will allow the well to be capped and also facilitate retrieval of the tubing during later sampling events.
    c. Use a length of rope or string to tie the tubing to the well cap.
    d. Alternatively, if tubing and safety line are to be saved and reused for sampling the well at a later date, coil the tubing neatly and placed in a clean plastic bag that is clearly labeled with the well ID ensuring the bag is tightly sealed before placing it in storage.
28. Secure the well and properly dispose of personal protective equipment (PPE) and disposable equipment.
29. Complete the procedures for packaging, shipping, and handling with the associated Chain-of- Custody.
30. Complete decontamination for flow-through analytical cell and submersible or bladder pump, as appropriate *(TGI – Groundwater and Soil Sampling Equipment Decontamination).*
31. At the end of each day of the sampling event, perform calibration check of field instruments and record procedure and results in field log.

9 Waste Management

Materials generated during groundwater sampling activities, including disposable equipment and excess purge water, will be stored on site in appropriately labeled containers and disposed of properly. Waste will be managed in accordance with the *TGI – Investigation-Derived Waste Handling and Storage*, the procedures identified in the

FIP or QAPP as well as state-, federal- or client-specific requirements. Be certain that waste containers are properly labeled and documented in the field logbook.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Management of the original documents from the field will be completed in accordance with the site- specific QAPP.

In general, forms (e.g., Low-Flow Sampling Field Forms), logs/notes (including daily field and calibration logs), digital records, and Chain-of-Custody records will be maintained by the field team lead.

Field logs and Chain-of-Custody records will be transmitted to the Arcadis Project Manager and/or Task Manager, as appropriate, at the end of each day unless otherwise directed. Electronic data files will be sent to the project team and uploaded to the electronic project folder daily.

Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements.

11 Quality Assurance

Quality assurance procedures shall be conducted in accordance with the Arcadis Quality Management System or the site-specific QAPP.

Unless described otherwise in the project-specific FIP/work plan, QAPP, or Sampling and Analysis Plan, quality assurance/quality control samples will be collected as follows:

- One duplicate for every 10 samples
- One laboratory matrix/matrix spike sample for every 20 samples
- In addition to the quality control samples to be collected in accordance with this TGI, the following quality control procedures will be observed in the field:
- Collect samples from monitoring wells, in order of increasing concentration, to the extent known based on review of historical site information if available
- Equipment blanks will include the pump and tubing (if using disposable tubing) or the pump only (if using tubing dedicated to each well)
- Collect equipment blanks after wells with higher concentrations (if known) have been sampled

- Operate all monitoring instrumentation in accordance with manufacturer's instructions and calibration procedures—calibrate instruments at the beginning of each day, verify the calibration at the end of each day, and record all calibration activities in the field notebook
- Clean all groundwater sampling equipment prior to use in the first well and after each subsequent well following the procedure for equipment decontamination

12 References

USEPA. 1986. *RCRA Groundwater Monitoring Technical Enforcement Guidance Document* (September 1986).

USEPA. 1991. *Handbook Groundwater, Volume II Methodology*, Office of Research and Development, Washington, DC. USEPN62S, /6-90/016b (July 1991).

USEPA Region I. 2017. *Low Stress (Low Flow) Purging and Sampling Procedures for the Collection of Groundwater Samples from Monitoring Wells (*EQASOP-GW4; September 19, 2017).

U.S. Geological Survey (USGS). 1977. *National Handbook of Recommended Methods for Water-Data Acquisition: USGS Office of Water Data Coordination*. Reston, Virginia.

13 Attachments

Attachment A – Low Flow Sampling Field Form

Attachment A

Low-Flow Sampling Field Form

GROUNDWATER SAMPLING FORM

Project No. _____ Well ID _____ Date _____

Project Name/Location _____ Weather _____

Measuring Pt. Description _____ Screen Setting (ft-bmp) _____ Casing Diameter (in.) _____ Well Material ____ PVC ____ SS

Static Water Level (ft-bmp) _____ Total Depth (ft-bmp) _____ Water Column (ft) _____ Gallons in Well _____

MP Elevation _____ Pump Intake (ft-bmp) _____ Purge Method: _____ Sample Method _____

Pump On/Off _____ Centrifugal _____ Submersible _____ Other _____

Sample Time: _____ Volume Purged _____

Purge Start _____ Gallons Purged _____ Sample ID _____ Sampled by _____

Purge End _____ Replicate/Code No. _____

Time	Minutes Elapsed	Rate (gpm)/(mL/min) 200mL/min +	Depth to Water (ft) -0.3	Gallons Purged	pH ± 0.1	Cond. (µMhos)/(mS/cm) ± 3%	Turbidity (NTU) ± 10%	DO (mg/L) ± 10%	Temp. (°C)/(°F) ± 3%	Redox (mV) ± 10mV	Appearance	
											Color	Odor
Stabilization Calculations (±)												
Stabilization Criteria					**± 0.1 s.u.**	**±3%**	**± 10% or within 1 NTU (1)**	**± 10%**	**±3%**	**±10 mV**		

(1) **Turbidity** < 50 NTU and ±10% or within 1 NTU of a previous reading when <10 NTU

Constituents Sampled	Container	Number	Preservative

Comments _____

Well Casing Volumes

Gallons/Foot					
	1" = 0.04	*1.5" = 0.09*	*2.5" = 0.26*	*3.5" = 0.50*	*6" = 1.47*
	1.25" = 0.06	*2" = 0.16*	*3" = 0.37*	*4" = 0.65*	

Well Information

Well Location: _____ Well Locked at Arrival: Yes / No

Condition of Well: _____ Well Locked at Departure: Yes / No

Well Completion: Flush Mount / Stick Up Key Number To Well: _____

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TGI - Manual Water-Level and NAPL Monitoring

Rev: 5

Rev Date: March 8, 2024

Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
0	0	October 11, 2018	All	Updated and re-written as TGI	Marc Killingstad Everett H. Fortner III
1	1	May 8, 2020	All	Updated and added NAPL gauging	Marc Killingstad Everett H. Fortner III Andy Pennington
2	2	April 5, 2022	All	Formatting and Revisions	Martha Wulftange
3	3	December 22, 2022	11	Data Management	Martha Wulftange
4	4	March 1, 2023	All	Annual review completed by SME. Document Revision Number and Document Date Updated.	Everett H. Fortner III
5	4	March 8, 2028	All	Annual review completed by SME. Document Revision Number and Document Date Updated.	Everett H. Fortner III

Approval Signatures

Prepared by: 

3/8/2024

Everett H. Fortner III, PG (Preparer) — Date

Reviewed by: 

3/8/2024

Marc Killingstad, PE (Subject Matter Expert) — Date

1 Introduction

This TGI describes the equipment, field procedures, materials, and documentation procedures to measure and record water-levels using an electronic water-level probe or an oil-water level indicator. This TGI also describes procedures for measuring in-well thicknesses of non-aqueous phase liquid (NAPL), both light and/or dense (LNAPLs and DNAPLs, respectively).

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

The objective of this Technical Guidance Instruction (TGI) is to describe procedures to measure and record water-levels (groundwater and surface-water) using manual water-level meters. Water levels may be measured using an electronic water-level probe or an oil-water level indicator from established reference points (e.g., top of casing). Reference points must be surveyed to evaluate fluid level elevations relative to a vertical datum (e.g., North America Vertical Datum of 1988 [NAVD88] relative to sea level). This TGI also describes procedures for measuring in well thickness of NAPL and DNAPLs.

Surface water-levels can be measured from stilling wells or fixed points (bridges, walls, etc.) and measuring from an established point of reference using a water-level meter. In some cases, surface water water-levels may be determined from a graduated stream gauge, attached to a pole located in open water with known elevation, without the use of a water-level meter.

The use of pressure transducers or other automated devices for the collection of groundwater elevation data will be subject of *TGI – Water-Level Monitoring using Pressure Transducers and TGI – Water-Level Measurements using Sonic Meters.*

4 Personnel Qualifications

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as specified in the Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. In addition, Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The HASP and other documents will identify other training requirements or access control requirements.

5 Equipment List

The following field equipment is suggested for water-level measurements:

- Site-specific Health and Safety Plan (HASP).
- Appropriate personal protective equipment (PPE) as specified in the HASP.
- Electronic water-level indicator graduated in 0.01 ft. increments.
- Electronic oil-water (interface) level indicator graduated in 0.01 ft. increments, if necessary.
- Non-phosphate laboratory soap (Alconox or equivalent), brushes, clean buckets or clean wash tubs.
- Distilled or de-ionized (required for some sites) water for equipment decontamination.
- Photoionization detector (PID) and/or organic vapor analyzer (optional).
- 150-foot measuring tape (or sufficient length for the maximum site depth requirement) – if required for total depth measurements of deeper wells.
- Solvent (methanol/acetone/isopropyl alcohol) rinse – optional.
- Spray bottle for solvent - optional
- Plastic drop cloth (e.g. Weatherall Visqueen) to place beneath the buckets or tubs to reduce potential for contamination of the tape or probe.
- Tools and/or keys required for opening wells.
- Well construction summary table and/or well construction logs.
- Summary table of previous water-level measurements.
- Field notebook and/or smart device (phone or tablet) or appropriate field forms (see Attachment 1).
- Indelible ink pen

6 Cautions

Electronic water-level indicators and oil-water interface probes may sometimes produce false-positive readings. For example, if the inside casing surface of the well or stilling tube has condensation above the water level, then an electronic water-level probe may produce a signal by contacting the sidewall of the well, rather than the true water-level surface. For accuracy, the electronic water-level probe and/or interface probe should be raised and lowered several times at the approximate depth where the instrument produces a tone indicating a fluid interface to verify consistent, repeatable results (three or more times). Additionally, some wells may be constructed with a sump. If local/regional groundwater levels have declined such that the water-level is below the base of the well screen, a sump may still contain water and provide an erroneous measurement. Therefore, possessing and comparing measurements with a well construction summary table or well construction log is recommended for proper reporting.

If the presence of a NAPL is known or suspected within specific wells, do not use an electronic water- level indicator. Use an oil-water interface probe instead. If NAPL presents ignition or explosion hazards, an intrinsically safe oil-water interface probe is required to be used with grounding and following the manufacturer's instructions.

If the NAPL is known to be very viscous or problematic to gauge, the data quality will require additional consideration prior to measuring. Staff will consider the data quality objectives for the gauging activity – e.g., if quantifying NAPL thickness is necessary, or if assessing the presence/absence is sufficient.

Alternate NAPL measurement methods (such as using drop pipes or temporary coatings for down-well equipment) may be considered.

When measuring total well depths with an electronic water-level indicator, the measurement must have a correction factor applied for post processing or completed at the time of measurement that is equal to the length of the probe beneath the circuit closing electrodes (if applicable to the instrument). This is necessary because the tape distance markings are referenced to the electrode, rather than the end of the probe. Some newer instruments do not have an offset electrode and this correction factor is needed. In addition, total depth measurements are difficult with wells that have large water columns due to buoyancy issues. In addition, the total depth measurement will include notes that indicate a soft or hard bottom if recognized during the measurement.

Ensure that the type of electronic water-level indicator is compatible with the depth and diameter of the wells to be measured. Some smaller piezometers or larger diameter well stilling tubes will accommodate only smaller diameter probes.

7 Health and Safety Considerations

The HASP will be followed, as appropriate, to ensure the safety of field personnel. Access to wells may expose field personnel to hazardous materials such as contaminated groundwater or oil. Other potential hazards include pressurized wells, stinging insects that may inhabit well heads, other biologic hazards (e.g. ticks in long grass/weeds around well head), and potentially the use of sharp cutting tools (scissors, knife). Appropriate personal protective equipment (PPE) will be worn during these activities. Only use non-toxic peppermint oil spray for stinging insect nests. Open well caps slowly and keep face and body away to allow to vent any built-up pressure. Field personnel will thoroughly review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives.

Obtaining measurements from active pumping wells requires knowledge of the construction and design, as the indicator probe and tape can become intertwined within down-well equipment (such as pump impellers) causing a serious health and safety hazard and equipment damage. Ensure that stilling wells have a perforated end and capped bottom to inhibit tape from extending into the downhole pump depth. If a stilling tube is not present or the still tube construction is not known, determine a conservative "not to exceed" measurement depth based on the top of pump depth with an added safety factor. If all information is not known, a water-level will not be taken from the pumping well until clarification on depths are available.

8 Procedure

Calibration procedures and groundwater level measurement procedures for electronic water-level indicators and oil-water indicators are described in the sections below. Calibration documentation can berequested from the rental or manufacturer.

Calibration Procedures

If the indicator requires length and markings verification is required by project data quality plan or other reasons, then the following steps may be used:

- Measure the lengths between each increment marker on the indicator with a measuring tape. The appropriate length of indicator measuring tape, suitable to cover the depth range for the wells of interest, will be checked for accuracy.
- If the indicator measuring tape is inaccurate, the probe will require to be sent back to the manufacturer or rental company. If a replacement can't immediately be available, then an offset can be measured to correct the measurements.
- If multiple water-level indicators and/or oil-water interface probes are being used for an event, calibration of the multiple devices will be required by measuring a water-level at a single well contemporaneously with all indicators to be used and calculated correction factors provided for data processing (typical corrections are small and range from 0.01 to 0.03 foot).
- Equipment calibration will be recorded in the field logbook and/or smart device.

Water-Level Measurement Procedures

The general procedures to be followed for the collection of fluid level measurements and well depths from the monitoring wells are as follows:

- Check that the water-level/oil-water level indicator battery is functional, before mobilization and prior to each work day (e.g., turn power on and check that meter sounds when probe is lowered into a bucket of water – note that water-level meters will not work with low-electrical-conductivity liquids such as distilled water).
- Record instrument make, model, serial number, and (if present) Arcadis ID number in the field form or electronic field form.
- Don disposable nitrile gloves. Decontaminate the water-level/oil-water indicator, any attached tape and the spool with laboratory-grade soap and distilled water (see Initial Decontamination Procedures below). The spool requires caution with cleaning as it is not water-proof and can be damaged during cleaning.

- The top of the monitoring well will be cleaned with a clean rag to prevent loose particulate matter from falling into the well.
- Perform a well inspection (note that a well inspection form may be required to be filled out along with a photo to document the conditions).
- Place clean plastic sheeting on the ground next to the well.
- Unlock and/or open the monitoring well cover while standing upwind from the well (note that some wells may be under pressure and precaution should be taken with opening well caps – see Section 6).
- Measure the volatile organics present in the monitoring well head space with a PID and record the PID reading (if applicable and requirement for the site).
- Allow the water-level in the well to equilibrate with atmospheric pressure for a few minutes (check previous field forms or field books for equilibration time, if noted).
- Locate the measuring reference point that correlates to the survey point on the well casing. If one is not found, make a reference point by notching the highest and/or north point on the inner casing (or outer if an inner casing is not present) or mark with a permanent mark. All downhole measurements will be taken from the reference point. Document any changes or new reference point addition.
- Measure to the nearest 0.01 foot and record the height of the inner well casing and outer protective casing to ground level (note that some well pads are raised and are not at true ground surface).
- Lower the indicator probe into the center of the well until contact with the water surface is indicated by either an audible alarm or light. The sensitivity of the probe may need adjustment if the alarm or light is not strong signal. Use and install a tape guide (available from some manufacturers) to help with accuracy and provide protection with damaging the measurement tape. If a tape guide is not available, make sure that the tape does rub on the inner or outer casing which could fray and damage the tape.
- If an oil-water interface probe is being used to measure depth and thickness of NAPL, lower the interface probe into the center of the well until a contact with the NAPL surface is indicated by either audible alarm or light. The sensitivity of the probe may need adjustment if the alarm or light is not strong signal. To gauge the water level in a well which contains LNAPL (LNAPL-water interface), advance the interface probe past the LNAPL-water interface until the probe produces a solid audible alarm indicating water. While slowly retrieving the probe upward, the equipment will produce a different tone when the LNAPL-water interface is reached (typically this is a multiple alarm sound or flashing light). This level should represent the depth to water. The depth indicating the bottom of the water column and top of DNAPL layer, if any, is indicated by the multiple alarm signal or flashing light emitted by the interface probe.
- Hold the tape at the measuring point and repeat the measurement two more times.
- Read and record measurement to the nearest 0.01 foot. Check the measurement with previous measurements, if available, and note any anomalies/discrepancies; if significant, contact the project staff.

- Measure and record total depth of well (see Total Depth Measurement Procedures below); note that measurement of total depth is not always performed at wells containing LNAPL or DNAPL, in order to reduce decontamination of the instrument and reduce potential exposure to NAPL.
- Record all measurements (with date and time collected to the nearest minute) and note any inconsistencies/anomalies and relevant observations in the field notebook and/or smart device or appropriate field forms.
- Follow decontamination procedure outlined below before measuring subsequent wells (see
- Decontamina*tion after Water Level and Total Depth Measurements* below).
- Replace cap and lock the well when all activities are completed.

Total Depth Measurement Procedures

- Weighted tape or electronic water-level indicator can be used to measure the total well depth.
- Follow initial procedures noted above in Water-Level Measurements above.
- Lower indicator probe (or tape) until weighted end is resting on the bottom of the well. Raise indicator slowly until there is no slack in the tape. Gently estimate the bottom of the well by slowly raising and lowering the indicator: great care should be taken to avoid damaging the sensor on the probe. The operator may find it easier to allow the weight to touch bottom and then detect the 'tug' on the tape while lifting the weight off the well bottom.
- Because of tape buoyancy and weight effects encountered in deep wells with long water columns, it may be difficult to determine when the probe is in contact the bottom of the well and sediment in the bottom of the well can also make it difficult to determine total depth. Care must be taken in these situations to ensure accurate measurements.
- If total depth measurements are to be collected during low-flow sampling events, the measurement will be made only after low-flow sampling has been completed or at least 12 hours prior to initiating sample collection from the well, in order to minimize: 1) mixing of the stagnant water at the top of the well column with potential formation water underneath; and/or 2) agitation and subsequent entrainment of possible sediment collected at the well bottom).
- Read and record measurement to the nearest 0.1 foot. Please refer to the note regarding total depth measurements described in Section 5 Cautions above.
- Follow decontamination procedure outlined below before gauging the next well (see Decontaminationafter *Water Level, NAPL Level, and Total Depth Measurements* below).

Initial Decontamination

- Note that there may be project specific decontamination procedure documents that will be followed in lieu of the below procedures.
- Set up a decontamination station consisting of three clean buckets (e.g., 5-gallon buckets). The buckets should not be used to containerize purge water; they will be used for decontamination purposes only.
- Fill the first bucket with one gallon of distilled water (use deionized water if metals are a contaminant at the site) and add non-phosphate laboratory-grade soap. Fill the second bucket

with distilled water (use deionized water if metals are a contaminant at the site) and leave the third bucket empty. Place the drop cloth underneath.

- Unwind the entire tape from the spool into a bucket with non-phosphate laboratory-grade soap and distilled water; Brush the tape carefully to remove dirt and possible contamination, using a brush dedicated to the wash bucket.
- Carefully brush all dirt of the spool and wipe down with a soapy cloth or paper towel.
- Transfer the tape into the second bucket containing rinse water. Carefully brush the tape using a second brush, dedicated to the rinse bucket. Lift the tape out of the bucket and allow rinse water to drip off the tape.
- Transfer the tape to the third bucket. Wind the tape onto the spool while wiping excess water off the tape using a paper towel.

Decontamination after Water Level, NAPL Level, and Total Depth Measurements

- Set up a decontamination station consisting of three clean buckets, fill according to the initial decon procedure.
- Unwind the only the length of tape used for gauging from the spool into a bucket with laboratory-grade soap and distilled water. Brush the tape carefully to remove dirt and possible contamination, using a brush dedicated to the wash bucket.
- Continue as described above.
- Extra care should be taken to clean the probe after a total depth measurement. All sediment or dirt needs to be removed during decontamination.
- If an oil-water interface probe is used to gauge NAPL, a solvent may be necessary to remove all NAPL residue. After decontaminations steps above, use a spray bottle filled with chosen solvent (ex. isopropyl alcohol) and spray across all surfaces of the tape. Use paper towels to wipe off solvent and/or residue. This step may be repeated if necessary.

Notes:

- Collect equipment blanks if required by the work plan (minimum 1 per 20 samples or 1 per sampling event).
- Prepare new wash solution and rinse water when necessary (e.g., every 10 to 20 wells). The spent wash and rinse solution should be discharged according to site practices.
- The decontamination station may be expanded by adding extra rinse and/or detergent stations (i.e., solvent wash station) to the set up. The addition of more stations depends on the requirements of the work plan or the site-specific Field Sampling and Quality Assurance Plan and outlined in the project field plan or kick-off meeting.
- Small crates or washtubs are a possible substitute for the buckets. In any case, it is recommended to use containers with a lid.

9 Waste Management

Decontamination fluids, PPE, and other disposable equipment will be properly stored on site in labeledcontainers and disposed of properly. Be certain that waste containers are properly labeled and documented in the field log book. Review *TGI – Investigation Derived Waste Handling and Storage*, foradditional information and state- or client-specific requirements.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Field personnel will complete all applicable field forms for each test (see attached forms and FieldNow® available forms). Forms will include recommended data file naming protocol per the field implementation plan. Data that has been collected must be transmitted at the end of the day (notes/forms/data file). The files must be transmitted to the appropriate project directory (via direct transfer or email to data manager). Work completed that day and any relevant observations noted during the daily activities as well as copies of the data mentioned above should be summarized and provided in an email. The appropriate team member will review the data for accuracy and provide feedback. Field equipment calibration, decontamination activities, and waste management activities will be recorded in the field notebook, appropriate field form, or daily log.

Refer to the Quality Procedure Data Management, QP 4.09, for additional information on data management.

11 Quality Assurance

Suggested quality control measures are below; project teams may implement some or all of these at their discretion and based on project data quality needs.

- As described in the detailed procedure, the electronic water-level meter and/or oil-water interfaceprobe can be calibrated prior to use versus an engineer's rule to ensure accurate length demarcations on the tape or cable. The results will be recorded.
- Measurements will be completed three times, with the final measurement recorded.
- Fluid interface measurements will be verified by gently raising and lowering the instrument through each interface to confirm repeatable results.
- Field notes will be reviewed by the project team once the field data has been delivered.

12 References

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U.S. Environmental Protection Agency, 2013. *SESD Operating Procedure, Groundwater level and Well Depth Measurement.* January 29.

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TGI – Monitoring Well Development

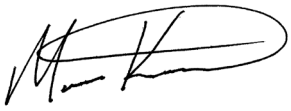
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	0	4/24/2017	All	Re-written as TGI	Marc Killingstad
	1	4/12/2022	All	Updated to new format and some minor content changes	Marc Killingstad
	2	4/5/2023	All	Annual review completed by Marc Killingstad. Updated document revision number and date, version control and signature page.	Marc Killingstad

Approval Signatures

Prepared by:		4/5/2023
	Jay Erickson (Preparer)	Date
Reviewed by:		4/5/2023
	Marc Killingstad (Subject Matter Expert)	Date

1 Introduction

This Technical Guidance Instruction (TGI) covers the development of screened wells used for obtaining representative groundwater information and samples from granular aquifers (i.e., monitoring wells).

Note: This TGI only applies to monitoring well development and not remediation (injection/extraction) well development.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

The objectives of monitoring well development are:

1. Repair damage to the borehole wall from drilling that can include clogging, smearing or compaction of aquifer materials;
2. Remove fine-grained sediment from the formation and filter pack that may result in high turbidity levels in groundwater samples;
3. To re-sort formation and filter pack material adjacent to the well screen;

4. To recover any drilling fluids (if used) that may affect the permeability of the formation and filter pack or alter the water quality around the well; and
5. To optimize the well efficiency and hydraulic communication between the well screen and the formation.

Successful monitoring well development is dependent on the following:

1. Hydrostratigraphy – Permeable formations containing primarily sand and gravel are more easily developed due to lower percentages of silt and clay material. Water in permeable formations can be moved in and out of the screen and/or through the formation easier than in less permeable deposits.
2. Well Diameter – Development tooling including brushes, surge blocks, pumps and jetting tools are more readily available for wells 4 inches in diameter and greater.
3. Well Design – Wells with filter packs and screens designed to match the formation through the analysis of formation sieve samples are easier to develop. An important aspect to well design is to minimize the size of the annular space between the formation and well screen. Adequate room must be allowed for the proper installation of well materials, but not too large as to prevent/reduce communication with the surrounding formation.
4. Drilling Methods – Different drilling methods result in varying amount of borehole damage and, therefore, impact the degree to which development will be successful.

Well development methods for monitoring wells include the following:

1. Bailing – Use of a bailer to remove water and sediment from the well casing. This technique does little to remove fines from the filter pack and may lead to bridging of sediment since the flow is in only one direction, toward the well screen. The most effective use of bailing during monitoring well development is in conjunction with other methods (e.g., surging/swabbing) to remove fines accumulated in the monitoring well between cycles of other development methods.
2. Pumping/over pumping – Use of a pump to remove water and sediment from the well casing, over pumping involves pumping the well at a rate that exceeds the design capacity of the well. Similar to bailing, this technique does little to remove fines from the filter pack and may lead to bridging of sediment since the flow is in only one direction, toward the well screen. Small diameter monitoring wells have the additional constraint on pump size and flow rates which further limit the effectiveness of this methodology.
3. Backwashing (rawhiding) – Consists of starting and stopping a pump intermittently to produce rapid pressure changes in a well. This method can produce better results than pumping alone since the procedure involves movement of the water in and out of the screen and formation. However, in many cases the surging action is not rigorous enough to fully develop the well and might be considered the final phase of development after a more rigorous method has been used. Again, small diameter monitoring wells have the additional constraint on pump size and flow rates which further limit the effectiveness of this methodology.
4. Surging/swabbing – Use of a mechanical surge block or swabbing tool to operate like a piston with an up and down motion. The downstroke causes a backwash action that breaks up bridged sediment and the upstroke pulls the dislodged sediment into the well. This method works well for both small and large diameter monitoring wells. Care should be taken on the downstroke so as not to force fines back into the formation, frequent pumping/purging during surging help to keep fines out of the well. Double surge blocks are recommended, and this is typically the most effective method for development of monitoring wells.

5. Jetting – Use of a tool fitted with nozzles that direct streams of water horizontally into well screens at high velocity. Due to the size of the tooling, this method is better suited for wells 4 inches in diameter and larger. The method is also more effective with wire-wrapped/continuous slot screens due to the increased open area. Jetting requires specialized equipment and concurrent pumping to prevent reintroducing fines into the filter pack. Additionally, depending on the configuration of the tool, jetting may require subsequent surging/pumping to remove fines dislodged in the filter pack and formation. Typically, jetting is not a preferred option for new well development but may be effective as part of a re-development/rehabilitation effort.

For most situations, surging/swabbing coupled with bailing or pumping to remove dislodged materials is recommended.

Final well development for properly designed and constructed monitoring wells may begin after the annular seal materials have been installed and allowed to cure, since these wells are designed to retain approximately 90% of the filter pack material. This cure time is typically at least 24 to 48 hours after the sealing materials have been installed.

This TGI is meant to provide a general guide for proper development of newly installed monitoring wells.

A site-specific field implementation plan (FIP) for well installation and development detailing the specific methods and tools is strongly recommended to provide site-specific instruction and guidance.

4 Personnel Qualifications

Generally, Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, and/or state/federal regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as specified in the Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. In addition, Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The HASP and other documents will identify other training requirements and access control requirements.

The designated Field Manager is responsible for periodic observation of field activities and review of field generated documentation associated with this TGI. The Field Manager is also responsible for implementation of corrective action if problems occur (e.g., retraining personnel, additional review of work plans and TGIs, variances to QC sampling requirements, issuing non-conformances, etc.).

Prior to mobilizing to the field, personnel will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP)/field sampling plan/work plan, Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Field personnel assigned to install and develop monitoring wells are responsible for completing their tasks in accordance with the specifications outlined in this TGI and other appropriate and relevant guidelines.

Monitoring well development activities will be performed by persons who have been trained in proper well development procedures under the guidance of an experienced field geologist, engineer, or technician.

5 Equipment List

Required equipment depends on the selected method and should be detailed in the site-specific FIP; however, the following are typically required.

- Approved site-specific Health and Safety Plan (HASP)
- Approved site-specific FIP which will include site map, well construction information/borehole information, and development plan
- Personal protective equipment (PPE) and health and safety equipment, as required by the HASP
- Field notebook and/or smart device (phone or tablet)
- Cleaning/decontamination equipment
    - Non-phosphate laboratory soap (Alconox or equivalent), brushes, clean buckets or clean wash tubs—new buckets or tubs will be purchased if it cannot be determined if the presentitems are clean
    - Distilled or de-ionized water for equipment decontamination
- Monitoring well keys
- Water-level meter
- Down-hole multiparameter water quality sonde (e.g., YSI)
- Plastic sheeting (e.g., Weatherall Visqueen) to protect all down-hole sampling equipment fromcontact with potential sources of contamination
- Well development forms/logs
- Well construction logs/diagrams
- Weighted tape (of sufficient length for maximum site depth)
- Turbidity meter
- Camera
- Watch/timing device

6 Cautions

Different USEPA regions and/or state regulatory agencies may stipulate deviations from this document. It is the responsibility of the Project Team (Project Manager and Technical Lead) to be fully aware of the requirements from the applicable regulatory framework.

Prior to beginning field work, the project technical team will ensure that all field logistics (e.g., access issues, health and safety issues, communication network, schedules, etc.) and task objectives are clearly understood by all team members. An internal call with the project technical team to review the FIP/field sampling plan/work plan scope and objectives is strongly recommended prior to mobilization to ensure that the field work will be effectively and efficiently executed.

Where surging is performed to assist in removing fine-grained material from the sand pack, surging must be performed in a gentle manner. Excessive suction could promote fine-grained sediment entry into the outside of the sand pack from the formation.

Avoid using development fluids or materials that could impact groundwater or soil quality or could be incompatible with the subsurface conditions.

In some cases, it may be necessary to add potable water to a well to allow surging and development, especially for new monitoring wells installed in low permeability formations. Before adding potable water to a well, the Certified Project Manager (CPM) and/or Project Hydrogeologist must be notified, and the CPM shall make the decision regarding the appropriateness and applicability of adding potable water to a well during well development procedures. If potable water is to be added to a well as part of development, the potable water source should be sampled and analyzed for constituents of concern, and the results evaluated by the CPM prior to adding the potable water to the well. If potable water is added to a well for development purposes, at the end of development the well will be purged dry to remove the potable water, or if the well no longer goes dry then the well will be purged to remove at least three times the volume of potable water that was added

7 Health and Safety Considerations

Field activities associated with monitoring well development will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities.

Appropriate PPE will be worn at all times in line with the task and the site-specific HASP.

Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work.

Access to well locations may expose field personnel to hazardous materials such as contaminated groundwater or NAPL (e.g., petroleum hydrocarbons, chlorinated solvents). Other potential hazards include pressurized wells, stinging insects that may inhabit well heads, other biological hazards (e.g., ticks in long grass/weeds around wellhead), and potentially the use of sharp cutting tools (scissors, knife). Open well caps slowly and keep face and body away while allowing to vent any built-up pressure to vent. Only use non-toxic peppermint oil spray for stinging insect nests. Review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives and use appropriate hand protection.

Do not enter confined spaces unless following appropriate confined space entry procedures specified in the HASP.

If thunder or lightning is present, discontinue sampling until 30 minutes have passed after the last occurrence of thunder or lightning.

8 Procedure

As indicated above, for most monitoring wells, gentle surging coupled with bailing or pumping to remove dislodged sediment is recommended.

8.1 Preliminary Well Development

After installation of the primary filter pack around the monitoring well screen, preliminary well development is recommended be performed to ensure that the filter pack settles and does not bridge within the annular space. The preliminary well development steps are as follows:

1. Measure and record depth to water, total depth of well, and depth to top of the sand pack in the annulus.
2. Use steel or weighted bailer to remove any fines that have accumulated in the bottom of the well.
3. Lower an appropriately sized double-surge block into the screened portion of the well on a rigid pipe or high-density tubing and gently cycle up and down to force water in and out of the screen slots and formation. A two-foot throw is recommended (use tape or chalk marks on the pipe or tubing); however, the entire length of well screen must be gently surged.
4. Start above the screen and gently surge over two-foot intervals while working down to the screen bottom.

NOTE: Care must be taken not to surge the well too aggressively at this point as the casing is not well-supported and damage could occur. The objective is to create enough surging action to settle the primary filter pack and provide some preliminary removal of accumulated materials before final development.

NOTE: If possible, ensure that the developer surges the block upward faster than downward to pull the fines out of the filter pack, instead of forcing them back in (and allowing for proper settlement).

5. Monitor the total depth of the well periodically during surging to ensure that we are not pulling excessive amounts of filter pack through the screen and remove any debris accumulated in the well with a weighted bailer or pump.
6. Re-measure the top of the sand in the annulus to see if more sand pack is necessary. Remove any fines that have accumulated out of the well using a submersible pump or weighted bailer.

NOTE: If the monitoring well was drilled using mud rotary drilling methodology or if significant fines were encountered during the well installation, consider adding a commercially available 'mud' dispersant (e.g., AQUA-CLEAR PFD, Nu Well 220, etc.) as part of the preliminary development. This will help to break up the 'skin' along the borehole wall created by either the drilling fluid or smearing during drilling and assist in final development. Follow manufacturer's directions for dosing, and the mixture should be worked through the entire saturated screen interval by gently surging or brushing.

8.2 Final Well Development

After sufficient time has passed to allow for proper curing of the well seal/grout (i.e., 24 to 48 hours), final well development can be performed. Final well development steps are as follows:

1. Don appropriate PPE (as required by the site-specific HASP).
2. Place plastic sheeting around the well.
3. Clean all equipment entering each monitoring well, except for new, disposable materials that have not been previously used.
4. Open the well cover while standing upwind of the well, remove well cap. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in

the field notebook. If the well headspace reading is less than 5 PID units, proceed; if the headspace reading is greater than 5 PID units, screen the air within the breathing zone. If the PID reading in the breathing zone is below 5 PID units, proceed. If the PID reading is above 5 PID units, move upwind from well for 5 minutes to allow the volatiles to dissipate. Repeat the breathing zone test. If the reading is still above 5 PID units, don the appropriate respiratory protection in accordance with the requirements of the HASP. Record all PID readings.

5. Obtain an initial measurement of the depth to water and the total well depth from the reference point at the top of the well casing. Record these measurements in the field logbook. It is recommended to use a weighted tape for the total well depth measurement.
6. The depth to the bottom of the well should be sounded and then compared to the completion form or construction diagram for the well. Any discrepancies should be reported immediately to the CPM and/or Project Hydrogeologist. If sand or sediment is present inside the well, it should first be removed by bailing. Do not insert bailers, pumps, or surge blocks into the well if obstructions, parting of the casing, or other damage to the well is suspected. Instead report the conditions to the CPM and/or Project Hydrogeologist and obtain approval to continue or cease well development activities.

NOTE: If the monitoring well was drilled using mud rotary drilling methodology or if significant fines were encountered during the well installation, it is recommended that a commercially available 'mud' dispersant (e.g., AQUA-CLEAR PFD, Nu Well 220, etc.) be included as part of the final well development to effectively break up the 'skin' along the borehole wall created by either the drilling fluid or smearing during drilling.

Per manufacturer's instructions, the general procedure for adding dispersant is as follows:

i. Determine volume of water in screen area and double the calculated volume to account for water in gravel pack and formation interface

ii. Once the water volume is determined, calculate the required treatment volume of dispersant need per manufacturer's recommendations

iii. Mix thoroughly before introducing into well

iv. The preferable application method utilizes a tremie line with the product applied into the screened area

v. Mixture should be thoroughly blended in well, then agitated via surging/swabbing/brushing repeatedly (e.g., every two hours) for a period of up to 24 hours

vi. The dispersant should sit for at least 6 to 8 hours or overnight before continuing well development activities

7. After allowing the dispersant to sit for the required time (if dispersant is used), start the mechanical development by lowering an appropriately sized double-surge block (or similar) into the well on a rigid pipe or high-density tubing.
    i. Surging should start above the screen to reduce the possibility of "sand-locking" the surge block. Initial surging should be with a long stroke and at a slow rate (20 to 25 strokes per minute)
    ii. After surging above the screen, the well should be cleaned via bottom-loading bailer, submersible pump, or inertia pump tubing with check valve to the bottom of the well

iii. Begin surging at the lower end of the screen, gradually working upward, surging in 2-ft intervals until the entire screen has been developed.

iv. Surge the well a minimum of 10 throws per 2-ft screen interval.

v. Each interval may require several surge cycles to achieve the best development.

vi. The entire length of well screen must be surged.

vii. Ensure that the developer surges the block upward faster than downward to pull the fines out of the filter pack, instead of forcing them back in (and allowing for proper settlement)

viii. measure total depth of the well periodically during surging to ensure that excessive amounts of sediment are not being pulled through the screen. Remove any debris accumulated in the well via simultaneous airlifting (if a combined tool is available) or with bailing/pumping.

8. After completing a cycle of surging, lower a bottom-loading bailer, submersible pump, or inertia pump tubing with check valve to the bottom of the well and gently bounce on the bottom of the well to collect/remove accumulated sediment, if any. Remove and empty the bailer, if used. Repeat until the bailed/pumped water is free of excessive sediment and contact at the bottom of the well feels solid. Alternatively, measurement of the well depth with a weighted tape can be used to verify that sediment and/or silt has been removed to the extent practicable, based on a comparison with the well installation log or previous measurement of total well depth.
9. After surging the well for a minimum of two cycles and removing excess accumulated sediment from the bottom of the well, re-measure the depth-to-water and the total well depth from the reference point at the top of the well casing. Record these measurements in the field log book.
10. Remove formation water by pumping/bailing.

    i. Where pumping is used, measure and record the pre-pumping water level.

    ii. Operate the pump at a relatively constant rate

    iii. Measure the pumping rate using a calibrated container and stopwatch, and record the pumping rate in the field log book

    iv. Measure and record the water level in the well at least once every 5 minutes during pumping

    v. Record any relevant observations in terms of color, visual level of turbidity, sheen, odors, etc.

    vi. Pump or bail until termination criteria specified in the site-specific FIP are reached

    vii. Record the total volume of water purged from the well

NOTE: The FIP may also specify a maximum turbidity requirement for completion of development. Unless otherwise specified the maximum turbidity should be 50 NTUs or less

11. While developing, take periodic water level measurements (at least one every five minutes) to determine if drawdown is occurring and record the measurements on the Well Development Log.
12. While developing, calculate the rate at which water is being removed from the well. Record the volume on the Well Development Log.
13. While developing, water is also periodically collected directly from the well or bailer discharge and readings taken of the indicator parameters: pH, specific conductance, and temperature. Development is

considered complete when the indicator parameters have stabilized (i.e., three consecutive pH, specific conductance, and temperature readings are within tolerances specified in the project work plans or within 10% if not otherwise specified), the extracted water is clear and free of fine sediment and most importantly, when acceptable volume of water has been removed and/or a sufficient amount of surging has been performed.

14. In certain instances, for slow recharging wells, the parameters may not stabilize. In this case, well development is considered complete when minimal amounts of fine-grained sediments are recovered, and an acceptable volume of water has been removed.
15. If the well goes dry, stop pumping or bailing. Note the time that the well went dry. After allowing the well to recover, note the time and depth to water. Resume pumping or bailing when sufficient water has recharged the well.
16. Contain all development water in appropriate containers.
17. When complete, secure the lid back on the well.
18. Place disposable materials in plastic bags for appropriate disposal and decontaminate reusable, downhole pump components and/or bailer

9 Waste Management

Investigation-Derived Waste (IDW), including purge water and decontamination liquids, will be stored on site in appropriately labeled containers and disposed of properly. Disposable materials will be stored and disposed of separately. Containers must be labeled at the time of collection and will include date, location(s), site name, city, state, and description of matrix contained (e.g., water, PPE). Waste will be managed in accordance with the *TGI – Investigation-Derived Waste Handling and Storage*, the procedures identified in the FIP/field sampling plan/work plan or QAPP as well as state-, federal- or client-specific requirements. Be certain that waste containers are properly labeled and documented in the field log.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

All well development activities will be documented on appropriate log forms as well as in a proper field notebook and/or PDA. Additionally, all documents (and photographs) should be scanned and electronically filed in the appropriate project directory for easy access. Pertinent information will include personnel present on site; times of arrival and departure; significant weather conditions; timing of well development activities; development

method(s); observations of purge water color, turbidity, odor, sheen, etc.; purge rate; and water levels before, during, and after pumping.

Management of the original documents from the field will be completed in accordance with the site-specific QAPP. Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements.

Development activities will be documented on appropriate field logs as well as in a proper field notebook. All field data will be recorded digitally or with indelible ink. Field forms, logs/notes (including daily field and calibration logs), digital records, and chain-of-custody records will be maintained by the field team lead. Any deviations or omissions from this TGI should be documented.

Initial field logs and forms will be transmitted to the Arcadis CPM and/or Technical Lead at the end of each day unless otherwise directed by the CPM. The field team leader retains copies of the field documentation.

11 Quality Assurance

Quality assurance procedures will be conducted in accordance with the Arcadis Quality ManagementSystem or the site-specific QAPP. Refer to the QAPP or FIP/sampling plan/work plan for specific requirements.

12 References

American Society for Testing Materials (ASTM), Designation D5521-05. *Standard Guide for Development of Ground-Water Monitoring Wells in Granular Aquifers*. American Society for Testing Materials. West Conshohocken, Pennsylvania.

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TGI – Monitoring Well Inspection Assessment

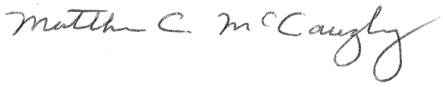
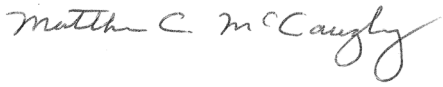
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Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
	0	4/19/2017	All	Re-written as TGI	Patrick Nolan M. McCaughey
	1	6/30/2022	All	TGI Title change from “Monitoring Well Integrity Survey” to “Monitoring Well Inspection Assessment” New Format	M. McCaughey

Approval Signatures

Prepared by:		6/30/2022
	Matthew C. McCaughey (Preparer)	Date
Reviewed by:		6/30/2022
	Matthew C. McCaughey (Subject Matter Expert)	Date

1. Introduction

This Technical Guidance Instruction (TGI) specifies the procedures for performing inspections and inventories of existing monitoring wells.

2. Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3. Scope and Application

Monitoring well inventories are periodically conducted to assess the condition of existing monitoring wells and to identify the need for repairs, replacement of parts, or replacement of wells that are no longer usable. A well inventory involves an inspection of the overall condition of the well, comparison of measurable quantities (e.g., riser stickup relative to grade and total depth), general verification of survey coordinates and elevation, and measurement of depth to water in the well.

This TGI applies to piezometers constructed analogous to monitoring wells. For simplicity, such piezometers are also referred to as monitoring wells for the remainder of this document. For all other types of wells (e.g., remediation wells such as injection, extraction, sparge, etc.), please refer to the appropriate guidance document regarding procedures for conducting inspections on those specific wells.

4. Personnel Qualifications

Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, the team will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP)/field sampling plan, Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Additionally, the Arcadis team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

The well inspection assessment procedures described below will be carefully adhered to and conducted under the supervision of an experienced geologist, engineer, or other qualified individual. Ideally, Arcadis personnel directing, supervising, or leading well assessment activities will have a minimum of one (1) year of field experience. It is recommended that field employees with less than six (6) months of experience be accompanied by a supervisor (as described above) to ensure that adequate survey techniques are employed.

The Arcadis CPM will be responsible for periodic observation of field activities and review of field generated documentation associated with this TGI and for implementation of corrective action if well conditions necessitate them.

5. Equipment List

The following materials will be available, as required, during performance of a monitoring well inventory:

- Health and safety equipment (as required by the site-specific Health and Safety Plan)
- Ruler or tape measure
- Water level indicator and/or interface probe
- Bailer
- Metal detector
- Indelible ink pen
- Paint pen
- Well keys
- Wrenches or ratchet set for accessing flush-mount well covers
- Cleaning equipment

- Well construction information (e.g., construction log, as-built, summary table, etc.)
- Digital camera (or phone with camera)
- Field notebook and digital data collection device (tablet or smartphone)
- Appropriate field form(s) (see Attachment A)

If feasible, a supply of typical replacement parts (e.g., locks, bolts, well caps) should be available to enable immediate usage, as necessary.

6. Cautions

It is important to confirm the correct identity of wells, particularly to those installed in a cluster. In these cases, however, the wells usually differ significantly in terms of depth below grade. During the well inspection assessment, verify that all wells are properly labeled by comparing their measured depth to the reported depth as installed. If the well identity is incorrectly labeled or not labeled, provide a clear, correct label using an indelible ink pen on the inside of the steel protective cover for the well, or on the outside of the steel protective cover using a paint pen. Take photos to document, as necessary.

One challenge with performing this task is locating existing monitoring wells in the field. Compilation and use of existing well records such as well location photos and aerial map images (e.g., Google Earth) are recommended. Note that the Well inspection Survey Assessment Form (**Attachment A**) includes a sketch area to help locate wells in future surveys.

If present, remove standing water from the curb box using a bailer before opening the monitoring well. Refer to project waste management plan for proper disposal of water.

7. Health and Safety Considerations

Field activities associated with monitoring well installation will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities. Care should be taken using tools to access flush-mount curb boxes. Wells in or near roadways may require a traffic control plan and traffic control measures (such as cones, flagging, and/or signs) prior to accessing. Access to wells containing chemicals of concern may pose a chemical exposure and biological hazard.

8. Procedure

The typical procedure for assessing the integrity of a monitoring well is outlined below.

8.1 Planning

Compile a list of wells to be inventoried and available information concerning their location and physical characteristics including photos and aerial maps.

8.2 Well Inspection Assessment

The well inspection procedure is described below:

- Locate the monitoring well using site maps and, if needed, a metal detector. Record field observations using the FieldNow application on digital device or an appropriate field form.
- Two field forms are provided in **Attachment A**. The first form is designed for a detailed description of an individual well inspection, while the second form is designed for the collection of multiple well inspections and observations on one page and can be used as a report summary table.
- Examine the well for the presence of an identification label. If absent, label the well with the appropriate well number after measuring the total depth of the well to verify that the depth matches the well number. If the well identity is incorrectly labeled or not labeled, provide a clear, correct label using an indelible ink pen on the inside of the steel protective cover for the well, and on the outside of the steel protective cover using a paint pen.
- Examine the surface condition of the well. Record the type of well (i.e., flush mount or above-grade stickup), condition of the well cover and surface seal. Confirm the protective casing is not bent or rusted through, the PVC casing is not broken or chipped, there is no evidence of frost heaving or subsidence.
- Unlock and open the well. Record the type (e.g., PVC or stainless steel), dimensions (i.e., casing diameter and riser stickup relative to grade), condition of the well casing, and type of well cap. Record any observations of recent modifications of the well casing. If the well cap is missing, replace with available parts or record the type of cap required.
- Measure the above-grade portion of the well riser stickup and compare to the known length of the stickup measured during well installation (surveyed top of inner casing elevation minus ground surface elevation). If the difference between the observed stickup length and the known stickup length is greater than 0.1 foot, the monitoring well location and elevation should be re-surveyed.
- Locate the marked measuring point along the top of the well casing. If no mark is visible, add a mark at the highest point of the casing using an indelible ink pen.
- Measure the depth to water, total depth of the well and any non-aqueous phase liquids (NAPL) thicknesses. For total depth measurements, account for any difference in calibration of the measuring tape on the probe (i.e., distance from part of probe that measures depth to water and the physical bottom of the probe that will measure total depth of the well). Record any obstructions encountered and a description of the feel of the well bottom (i.e., soft due to sediment or hard).
- Compare observations concerning the measured dimensions of the well with the listed values. Based on these results as well as other observations concerning the condition of the well, record any appropriate recommendations on the Well Inspection form (**Attachment A**).
- Perform any recommended maintenance activities that can be accomplished with available equipment.
- Remove all equipment from the well. If no additional maintenance activities are to be performed, close the well and collect all personal protection equipment (PPE) and other wastes generated for disposal.

8.3 Post-Assessment Activities

Depending on the results of the well inventory, several additional activities may be warranted prior to future usage of the well. Typical follow-up activities include replacement of missing parts, removal of sediment from the base of the well, re-surveying of the well, or complete replacement if the well is determined to be unusable.

As stated above, a supply of locks, bolts, and well caps should be available for immediate usage during performance of the well inventories. However, it may not be feasible to maintain a supply of all potential replacement parts due to the variety of well types in use. Therefore, a list of required replacement parts should be compiled during the performance of a well inventory event. At the conclusion of the event, the necessary replacement parts for all wells should be obtained and installed.

9. Waste Management

Materials generated during well inventory activities, including disposable equipment, will be disposed in appropriate containers.

10. Data Recording and Management

10.1 Digital Data Collection Process Overview

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

10.2 Digital Data Collection Tools for Well Inspections

Arcadis is transitioning from the use of paper forms to digital field forms using web based FieldNow applications accessible on field tablets and smart phones. Company-wide roll out of a FieldNow application for a Well Inspection app is targeted by the end of 2022. Paper forms are included in Revision 1 (June 2022) of this TGI. Specifically, a blank well inspection form is provided in **Attachment A**. Additional guidance and examples of the digital data collection tools for soil descriptions will be provided in the next revision to this TGI.

11. Quality Assurance

Field measurements will be double-checked periodically (e.g., at least one of these measurements per well should be repeated to verify accuracy).

12. References

No references apply to this TGI.

Attachment A

Monitoring Well Inspection Form

Monitoring Well Integrity Assessment Form

(For each item, check appropriate response or fill in the blank)

Date _____

Well ID _____ ID Clearly Marked? _____ Project Name _____

Photo filename _____ Project Number _____

Weather _____ Field Personnel _____

General Description of Surroundings _____

Well Condition:

Damaged? ☐ Abandoned? ☐
(Describe Below)
Stick Up ☐ Flush Mount ☐

Lockable cover? _____
Lock present? _____
Key number: _____

Stick up height _____
Casing material _____
Well diameter _____
Protective casing material: _____
Protective casing diameter: _____

Cap present? Type? _____
Vented? If so, how? _____

Measuring point clearly marked? _____
Total depth reported: _____
Total depth measured: _____
DTW: _____
Well obstructed? If so, depth? _____
Well bottom soft (sediment) or firm? _____

Flush Mount Wells Only

Gasket present? _____
Bolts present? _____
Teflon washers present?

Surface Condition:

Damaged? ☐
(Describe Below)

Pad/cement intact? _____
Curb box/well cover present? _____
Intact? _____

Seal condition _____
All bolts present? _____
Ground surface slopes away from well? _____

Location Sketch

Comments/Recommendations:

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TGI – Monitoring Well Installation

Rev: 1

Rev Date: June 23, 2022

Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
	0	4/24/2017	All	Re-written as a TGI	Marc Killingstad Peter C. Frederick
	1	6/23/2022	All	Put into new template format, reviewed and made minor revisions	Whitney Plasket Marc Killingstad

Approval Signatures

Prepared by:		4/7/2022
	Whitney Plasket	Date
Reviewed by:		4/7/2022
	Marc Killingstad	Date

1 Introduction

This Technical Guidance Instruction (TGI) describes methods used to install groundwater monitoring wells in unconsolidated aquifers. It is assumed that the monitoring well to be installed has been properly designed, including sizing of the filter pack and screen, the length of the screen, total depth of the well, material strength and compatibility and surface completion. Typical monitoring wells are constructed of manufactured screen and engineered filter pack and are generally suitable for formations with granular materials having a grain size distribution with up to 50% passing a #200 sieve and up to 20% clay-sized material. Monitoring wells installed in formations finer than this may not be able to produce turbidity free water.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

The monitoring well installation procedures set forth herein are consistent with the approach and methods presented in the American Society of Testing and Materials (ASTM) *D5092 – Standard Practice for Design and Installation of Groundwater Monitoring Wells* (ASTM D5092). As such, following this TGI in combination with proper well design (see appropriate TGI and/or consult with appropriate subject matter expert), well development (see appropriate TGI), groundwater sampling procedures (see appropriate TGI), and well maintenance and

rehabilitation (see appropriate TGI and/or consult with appropriate subject matter expert), will result in a monitoring well suitable for: (1) collection of groundwater samples representative of the surrounding formation and free of artificial turbidity; (2) measurement of accurate groundwater levels; and (3) hydraulic testing of formation sediments immediately adjacent to the open interval of the well to assess hydraulic properties (e.g., slug testing).

Monitoring well boreholes in unconsolidated (overburden) materials are often drilled using the hollow-stem auger drilling method; however, other drilling methods are also suitable for installing overburden monitoring wells and may be appropriate given site-specific geologic conditions or project objectives. These methods include drive-and-wash, spun casing, rotosonic (sonic), dual-rotary (Barber Rig), and fluid/mud rotary with core barrel or roller bit. Direct-push techniques (e.g., Geoprobe® or cone penetrometer) and driven well points may also be used in some cases within the overburden.

Monitoring wells to be installed within consolidated materials such as fractured bedrock are commonly drilled using air rotary, water-rotary (coring or tri-cone roller bit), or sonic drilling methods. For guidance when installing monitoring wells in consolidated materials, please consult the appropriate subject matter expert and, if available, the applicable guidance document.

The drilling method to be used at a given site will be selected based on site-specific consideration of anticipated drilling/well depths, site or regional geologic knowledge, type of monitoring to be conducted using the installed well, project objectives, and cost. Consultation with the appropriate subject matter expert is also strongly recommended.

No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools). No polyvinyl chloride (PVC) glue/cement will be used in constructing or retrofitting monitoring wells that will be used for water-quality monitoring.

Coated bentonite pellets are generally not recommended because of potential chemical incompatibilities between the coating material and groundwater chemistry.

Specifications of materials to be installed in the borehole will be obtained prior to mobilizing onsite. These materials generally include:

- Well casing (length, material, and diameter);
- Well screen (length, material, diameter, and slot size);
- Grout (typically neat cement grout, which is 5-6 gallons of water per 94 lb. bag of Portland Type I/II cement *with no bentonite* but, as applicable, up to 5% bentonite can be added);
- Filter pack (filter pack type and fine sand seal type, as applicable); and
- Bentonite (type, as applicable/needed, non-coated pellets or tablets are generally preferred over chips).

Well materials will be inspected and, if needed, cleaned, or replaced prior to installation. The field task manager or field team lead will communicate with the drilling company ahead of time to make sure the materials meet the required specification for well construction.

NOTE: If installing monitoring wells for per- and polyfluoroalkyl substances please refer to *TGI for Per- and Polyfluoroalkyl Substances (PFAS) Field Sampling Guide*.

4 Personnel Qualifications

Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or state/federal regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, Arcadis field personnel will review and be thoroughly familiar with relevant site-specific documents including but not limited to the field implementation plan (FIP)/task-specific work plan, Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis field personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Personnel responsible for overseeing drilling operations will have at least 16 hours of prior training overseeing drilling activities with an experienced geologist, environmental scientist, or engineer with at least 2 years of prior experience.

Arcadis personnel directing, supervising, or leading well installation activities will have a minimum of 1 year of previous environmental monitoring well installation experience. Field employees with less than six months of experience will be accompanied by a supervisor (as described above) to ensure that proper well installation techniques are employed.

Additionally, the Arcadis field team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization particularly the selected drilling method/rig.

Monitoring well installation activities will be performed by persons who have been trained in proper well installation procedures under the guidance of an experienced field geologist, engineer, or technician. Field sampling is typically performed for soil or bedrock characterization as part of monitoring well installation; therefore, field personnel will have undergone in-field training in soil or bedrock description and sample collection methods, as described in *TGI for Soil Drilling and Sample Collection, TGI for Bedrock Core Collection and Description, and TGI for Soil Description*.

5 Equipment List

The following materials may be required during soil boring and monitoring well installation activities:

- Site Plan with proposed soil boring/well locations;
- Field Implementation Plan (FIP)/Work Plan that includes site map with proposed well locations, well construction details (tabulated and drawings) which will include well casing material and size, well screen material and size, length of screen, target depth and screen interval, filter pack material, development methods, and previous boring logs (as available);
- Field Sampling Plan (FSP), and site-specific Health and Safety Plan (HASP);
- Personal protective equipment (PPE) as required by the HASP;

- Traffic cones, delineators, caution tape, and/or fencing as appropriate for securing the work area, if such are not provided by drillers;
- Appropriate soil sampling equipment (e.g., stainless steel spatulas, knife);
- Soil and/or bedrock logging equipment as specified in the FIP/work plan or other appropriate project documents;
- Appropriate sample containers and labels;
- Drum labels as required for investigation derived waste handling;
- Insulated coolers with ice, when collecting samples requiring preservation by chilling;
- Photoionization detector (PID) or flame ionization detector (FID);
- Ziplock style bags;
- Water level or oil/water interface meter;
- Locks and keys for securing the well after installation;
- Decontamination equipment (bucket, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels);
- Engineer's tape/measuring wheel;
- Weighted tape;
- Disposable bailers;
- Forms/notes:
    - Tablet with digital forms
    - Field notebook
    - Chain-of-custody forms
    - Digital camera (or smart phone with camera);
    - Appropriate field forms, consider including a photo of the well head and a Google Earth map showing the well location.

Prior to mobilizing to the site, Arcadis personnel will contact the drilling subcontractor or in-house driller (as appropriate) to confirm that appropriate sampling and well installation equipment will be provided. Specifications of the sampling and well installation equipment are expected to vary by project, and so communication with the driller is necessary to ensure that the materials provided will meet the project objectives. Equipment/materials typically provided by the driller could include:

- Drilling equipment required by the ASTM standard guidance document D1586, when performing split-spoon sampling;
- Disposable plastic liners (when drilling with direct-push equipment);
- Drums for investigation derived waste (IDW);
- Equipment to move IDW drums, if required;
- Drilling and sampling equipment decontamination materials;
- Decontamination pad materials, if required;

- Traffic cones, delineators, caution tape, and/or fencing as appropriate for securing the work area, if required; and
- Well construction materials.

6 Cautions

- Prior to beginning field work, underground utilities in the vicinity of the drilling areas will be delineated by the drilling contractor or an independent underground utility locator service. See Arcadis standard for proper utility clearance protocol.
- Prior to beginning field work, contact the project technical team (including Project Hydrogeologist) to ensure that all field procedures, logistics (e.g., access issues, health and safety issues, communication network, schedules, etc.), and objectives are clearly understood by all team members.
- Some regulatory agencies require a minimum annular space between the well or permanent casing and the borehole wall. When specified, the minimum clearance is typically 2 to 3 inches on all sides (e.g., a 2-inch diameter well requires a 6-inch diameter borehole). In addition, some regulatory agencies have specific requirements regarding grout mixtures and well seal materials. Determine whether the oversight agency has any such requirements prior to finalizing the drilling and well installation plan. If installing a monitoring well into consolidated sediments, refer to regulatory agency rules regarding casing.
- The maximum screen length may also be dictated by regulatory agencies. If installing a monitoring well with greater than a 10-ft screen, refer to regulatory agency rules regarding screen length.
- If dense non-aqueous phase liquids (DNAPL) are known or expected to exist at the site, refer to the project specific documents for additional details regarding drilling and well installation to reduce the potential for inadvertent DNAPL remobilization. Similarly, if light non-aqueous phase liquids (LNAPLs) are known or expected to be present as “perched” layers above the water table, refer to the *DNAPL Contingency Plan*. Follow the general provisions and concepts in the DNAPL contingency plan during drilling above the water table at known or expected LNAPL sites.
- Avoid using drilling fluids or materials that could impact groundwater or soil quality or could be incompatible with the subsurface conditions. Water used for drilling and sampling of soil or bedrock, decontamination of drilling/sampling equipment, or grouting boreholes upon completion will be of a quality acceptable for project objectives. Consider testing of water supply as necessary.
- Similarly, consider the compatibility between the well materials and the surrounding environment. For example, PVC well materials are not preferred when DNAPL is present. In addition, some groundwater conditions leach metals from stainless steel or are corrosive to metal well materials, and some remedial technologies are incompatible with certain materials of construction. If questions arise, contact the CPM and Project Hydrogeologist/Technical Lead to discuss.
- Specifications of materials used for backfilling the borehole will be obtained, reviewed, and approved to meet project quality objectives. Bentonite is not recommended where DNAPLs are likely to be present or in groundwater with high salinity. In these situations, neat cement grout is preferred.
- As noted above, coated bentonite pellets are not recommended for monitoring well construction, as the coating could impact the water quality in the completed well.

- Heat of hydration during neat cement grout curing must be considered to avoid damage to PVC well materials. The annular space for a typical monitoring well is small enough that heat of hydration should not create excessive temperature increases which may damage PVC well material. However, washouts in the borehole can lead to thick accumulations of grout which can produce enough heat during curing to weaken and potentially damage PVC casing. If heat of hydration is a concern, contact the Project Hydrogeologist/Technical Lead to address the issue.
- Similarly, it is imperative that backfill volumes (filter pack and well seal) be estimated and then closely monitored to ensure that materials are not 'lost' to the formation. If estimated volumes do not reasonably match actual volumes, contact the Project Hydrogeologist/Technical Lead to address the issue.

7 Health and Safety Considerations

Field activities associated with monitoring well installation will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities. The HASP may require that the drilling company provide their own HASP and/or Job Safety Analyses (JSAs).

The HASP will be followed, as appropriate, to ensure the safety of field personnel. Review all site-specific and procedural hazards as they are provided in the HASP, and review Job SafetyAnalysis (JSA) documents in the field each day prior to beginning work.

Prior to drilling, utility clearance must be performed (see Section 6). Appropriate personal protective equipment (PPE) must always be worn in accordance with the task and the HASP.

Working outside at sites with suspected contamination may expose field personnel to hazardous materials such as contaminated groundwater or NAPL (e.g., oil). Other potential hazards include biological hazards (e.g., stinging insects, ticks in long grass/weeds, etc.), and potentially the use of sharp cutting tools (scissors, knife). Only use non-toxic peppermint oil spray for stinging insect nests. Review client-specific health and safety requirements, which may preclude the useof fixed/folding-blade knives and use appropriate hand protection.

If thunder or lightning is present, discontinue drilling and sampling until 30 minutes have passed after the last occurrence of thunder or lightning.

8 Procedure

The procedures for installing groundwater monitoring wells are presented below:

Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Sonic, and Dual-Rotary Drilling Methods

1. Prior to monitoring well installation, determine the expected volumes of filter pack and seal materials including grout (neat cement or cement-bentonite) and bentonite (if applicable).
2. Locate boring/well location, establish work zone, and set up sampling equipment decontamination area.
3. During well installation, record construction details, measurements, and tabulate materials used (e.g., screen and riser footages; filter pack volume; bags of cement/sand; volume of grout; etc.) in the field notebook as well as appropriate field forms.

4. Advance boring to desired depth.
    a. Collect soil and/or bedrock samples at appropriate interval(s), document, and store samples for laboratory analysis as specified in the FIP/Work Plan.
    b. Decontaminate equipment between samples in accordance with the *TGI for Groundwater and Soil Sampling Equipment Decontamination* or if installing monitoring wells for per- and polyfluoroalkyl substances please refer to *TGI for Per- and Polyfluoroalkyl Substances (PFAS) Field Sampling Guide* for both sampling and decontamination guidance.
    c. A common sampling method that produces high-quality soil samples with relatively little soil disturbance is described in *ASTM D1586 – Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils* (ASTM D1586). Split-spoon samples are obtained during drilling using hollow-stem auger, drive-and-wash, spun casing, and fluid/mud rotary.
    d. Sonic drilling produces soil cores that, for the most part, are relatively undisturbed, but note that when drilling in consolidated or finer-grained sediment the vibratory action during core barrel advancement may create secondary fractures or breaks.
    e. Dual-rotary removes cuttings by compressed air or water/mud and allow only a general assessment of geology.
5. Describe each soil sample as outlined in *TGI for Soil Description* and document descriptions in the field notebook and/or field tablet or field forms and photo document the samples. It should be noted that electronic logs must be backed up and transferred to a location accessible to other project team members as soon as feasible to retain and protect the field data. During boring advancement, document all drilling events in field notebook or field forms, including blow counts (number of blows required to advance split-spoon sampler in 6-inch increments) and work stoppages. Blow counts will not be available if sonic, dual-rotary, or direct-push methods are used.
6. Before installing a screen, it is important to confirm that the borehole has been advanced into the targeted saturated zone. This is particularly important for wells installed to monitor the water table and/or the shallow saturated zone, as the capillary fringe may cause soils above the water table to appear saturated. If one or more previously installed monitoring wells exist nearby, use the depth to water at such well(s) to estimate the water-table depth at the new borehole location.

    NOTE: *To verify that the borehole has been advanced into the saturated zone, it is necessary to measure the water level in the borehole. For boreholes drilled without using water (e.g., hollow-stem auger, cable-tool, air rotary, air hammer), verify the presence of groundwater (and/or LNAPL, if applicable) in the borehole using an electronic water level meter, oil-water interface probe, or a new/decontaminated bailer. For boreholes drilled using water (e.g., drive and wash, spun-casing with roller-bit wash, sonic, or water rotary with core or roller bit), monitor the water level in the borehole as it re-equilibrates to the static level.*

    In low-permeability units like clay, fine-grained glacial tills, shale, and other bedrock formations, it may be necessary to wait overnight to allow the water level to equilibrate. Document depth to water in the borehole on the appropriate field forms and field notebook. If there are questions concerning the depth of the well/screen interval, consult with the project technical lead prior to finalizing well depth/screen interval. To the extent practicable, ensure that the depth of the well below the apparent water table is deep enough so that the installed well can monitor groundwater year-round, accounting for seasonal water-table fluctuations. When in doubt, err on the side of slightly deeper well installation.

7. Upon completing the borehole to the desired depth, if a screened well construction is required, install the monitoring well by lowering the screen and casing assembly through the augers or casing. Monitoring wells typically will be constructed of 2-inch-diameter (although sometimes 4-inch), flush-threaded PVC or stainless steel slotted or wire wrapped well screen and blank riser casing. Smaller diameters may be used if wells are installed using direct-push methodology or if multiple wells are to be installed in a single borehole, according to the well design as outlined in the FIP/Work Plan. The screen length and other construction details will be specified in the FIP/Work Plan based on regulatory requirements and specific monitoring objectives. Monitoring well screens are usually 5 to 10 feet long, but the screen length will depend on the purpose for the well and the objectives of the groundwater investigation and will (in most cases) be determined prior to the field mobilization.

   NOTE: *The slot size and filter pack gradation will be predetermined in the Work Plan (or equivalent) or FSP and based on site-specific grain-size analysis (sieve analysis) or other geologic considerations or monitoring objectives. Consult the Project Hydrogeologist and/or subject matter expert if there are questions/concerns regarding the filter pack and slot size specified. If the screen slot size and filter pack have not been based on site-specific grain-size analysis, consider collecting soil samples during well installation so future wells can be properly designed.*

   NOTE: *A blank sump may be attached below the well screen if the well is being installed for DNAPL recovery /monitoring purposes. If so, the annular space around the sump may be backfilled with filter pack during placement around the well screen.*

8. A blank riser will extend from the top of the screen to the level specified in the FIP/Work Plan (e.g., approximately 2.5 feet above grade if a stick up or just below grade where a flush-mounted monitoring well is specified).

   NOTE: *For wells greater than 50 feet deep, placement of centralizers may be desired to assist in centering the monitoring well in the borehole during installation. Refer to the FIP/Work Plan and/or consult with the Project Hydrogeologist/Technical Lead.*

9. When the monitoring well assembly has been set, using a tremie place the washed silica filter pack in the annular space from the bottom of the boring to a height above the top of the screen as specified in the FIP/Work Plan (typically placed to at least 2 feet above the top of the well screen). The filter pack will be placed, and drilling equipment extracted in increments until the top of the sand pack is at the appropriate depth.

   NOTE: *It is very important to verify that the expected volume of filter pack matches with the actual amount placed. There can be differences due to irregularities in the borehole geometry. Washout of the borehole will result in the need for greater than calculated well materials. If a difference of more than 10% is noted, consult with the Project Hydrogeologist/Technical Lead. The filter pack will be consistent with the screen slot size and the soil particle size in the screened interval, as specified in the FIP/Work Plan.*

10. After placement of the filter pack, preliminary well development is recommended be performed to ensure that the filter pack settles and does not bridge within the annular space and to remove any fines accumulated in the well during installation. This typically entails gently surging the entire well screen to prevent filter pack material bridging and to settle the filter pack prior to well seal installation. For recommended procedures, please refer to the *TGI for Monitoring Well Development*. Monitor the placement of the filter pack (e.g., with a weighted tape measure) and, as necessary during preliminary development (i.e., settlement), add filter pack to ensure proper thickness/height above screen is attained.

11. Depending on the project-specific requirements and applicable federal/state/local regulations, a well seal comprised of either fine sand or hydrated bentonite will then be placed in the annular space above the filter pack, typically at a minimum of 2 feet thick—follow the specifications outlined in the FIP/Work Plan). If non-hydrated bentonite is used, allow sufficient time for hydration to occur (typically a minimum of 30 minutes, but follow manufacturer's recommendations and/or specifications outlined in the FIP/Work Plan). Potable water may be added to hydrate the bentonite if the seal is above the water table. Monitor the placement of the fine sand/bentonite seal (e.g., with a weighted tape measure).

    NOTE: *Coated bentonite pellets are generally not recommended for monitoring well construction because of potential chemical incompatibilities between the coating material and groundwater chemistry.*

12. During the extraction of the augers or casing, a neat cement or cement/bentonite grout will be placed in the annular space from the well seal to a depth as specified in the FIP/Work Plan (e.g., approximately 2 ft. below groundwater surface). It is recommended that grout be placed with a tremie pipe. Ensure that seal materials are mixed at the proper ratios with water following manufacturer's recommendations.

    NOTE: *If it is necessary to install a monitor well into a permeable zone below a confining layer (i.e., confined conditions), particularly if the deeper zone is believed to have water quality that differs significantly from the zone above the confining layer, then a telescopic well construction may be considered.*

    In this case, the borehole is advanced approximately 3 to 5 feet into the top of the confining layer (depending upon the thickness of the confining layer), and a permanent casing (typically PVC or stainless steel) is installed into the socket drilled into the top of the confining layer.

    The casing is then grouted in place. The preferred methods of grouting telescoping casings include (1) pressure-injection grouting using an inflatable packer installed temporarily into the base of the casing, such that grout is injected out the bottom of the casing until it is observed at ground surface outside the casing; (2) displacement-method grouting (also known as the Halliburton method), which entails filling the casing with grout and displacing the grout out the bottom of the casing by pushing a drillable plug, typically made of wood to the bottom of the casing, following by tremie grouting the remainder of the annulus outside the casing; or (3) tremie grouting the annulus surrounding the casing using a tremie pipe installed to the base of the borehole.

    In all three cases, the casing is grouted to the ground surface, and the grout is allowed to set prior to drilling deeper through the casing. Refer to the FIP/Work Plan, Project Hydrogeologist, and/or subject matter expert for the completion of non-standard monitoring wells, including telescopic wells.

13. Install the monitoring well surface completion as specified in FIP/Work Plan. Typical completions are a locking, steel protective casing (extended at least 1.5 feet below grade and 2 feet above grade) over the riser casing and secure with a neat cement seal. Alternatively, for flush-mount completions, place a steel curb box with a bolt-down lid over the riser casing and secure with a neat cement seal. In either case, the cement seal will extend approximately 1.5 to 2.0 feet below grade and laterally at least 1 foot in all directions from the protective casing and will slope gently away from the casing to promote drainage away from the well.

14. When an above-grade completion is used, the riser will be sealed using an expandable locking plug and the top of the well will be vented by drilling a small-diameter (1/8 inch) hole near the top of the well casing or through the locking plug, or by cutting a vertical slot in the top of the well casing. When a flush-mount installation is used, the riser will be sealed using an unvented, expandable locking plug.

15. Monitoring wells will be labeled as specified in the FIP/Work Plan. If not specified, use indelible ink or paint with the appropriate designation on both the inner and outer well casings and/or inside of the curb box lid. If called for, mark a consistent measuring point by cutting a V in the PVC casing or marking the measuring point in black.
16. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section 9 below.
17. After completing well installation, finalize documentation and follow data management procedures outlined in Section 10 below.
18. For final well development guidance and procedures, please refer to the *TGI for Monitoring Well Development.*

Direct-Push Method

The direct-push drilling method may also be used to complete soil borings and install monitoring wells. Examples of this technique include the Diedrich ESP vibratory probe system, GeoProbe®, or AMS Power Probe® dual-tube system. Environmental probe systems typically use a hydraulically operated percussion hammer. Depending on the equipment used, the hammer delivers 140- to 350-foot pounds of energy with each blow and provides the force needed to penetrate very stiff to medium dense soil formations. The hammer simultaneously advances an outer steel casing that contains a dual-tube liner for sampling soil. The outside diameter (OD) of the outer casing ranges from 1.75 to 2.4 inches and the OD of the inner sampling tube ranges from 1.1 to 1.8 inches.

The outer casing isolates shallow layers and permits the unit to continue to probe at depth. The double-rod system provides a borehole that may be tremie-grouted from the bottom up. Alternatively, the inside diameter (ID) of the steel casing provides clearance for the installation of small-diameter (e.g., 0.75- to 1-inch ID) micro-wells.

If direct-push drilling has been determined to be a viable method for site conditions and project objectives, procedures for installing monitoring wells in soil using the direct-push method are described below.

1. Locate boring/well location, establish work zone, and set up sample equipment decontamination area.
2. Advance soil boring to designated depth, collecting samples at intervals specified in the FIP/Work Plan. Samples will be collected using dedicated, disposable, plastic liners. Collect and describe samples in accordance with the procedures outlined in Steps 4 and 5 above. Collect samples for laboratory analysis as specified in the FIP/Work Plan.
3. Upon advancing the borehole to the desired depth, install the micro-well through the inner drill casing. The micro-well will consist of approximately 1-inch ID PVC or stainless-steel slotted screen and blank riser. The filter pack, well seal, and neat cement/cement-bentonite grout will be installed as described, where applicable, in Steps 9 through 12 above.
4. Install surface completion (protective steel casing or flush-mount), as appropriate and as described in Steps 13 through 15 above.
5. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section 9 below.
6. After completing well installation, finalize documentation and follow data management procedures outlined in Section 10 below.

Driven Well Point Installation

If specified in the FIP/Work Plan, well points installed by pushing or driving using a drilling rig or direct-push rig (or hand-driven where possible) will typically consist of a 1- to 2-inch-diameter threaded steel casing with either 0.010- or 0.020-inch slotted stainless-steel screen. The screen length will vary depending on the hydrogeologic conditions of the site. The casings will be joined together with threaded couplings and the terminal end will consist of a steel well point. Because they are driven or pushed to the desired depth, well points do not have annular backfill materials such as sand pack or grout. Refer to the FIP/Work Plan and/or consult with the Project Hydrogeologist/Technical Lead and/or subject matter expert for specific guidance on drive point installation procedures/specifications.

9 Waste Management

IDW, including soil cuttings and excess drilling fluids (if used), decontamination liquids, and disposable materials (well material packages, PPE, etc.), will be placed in clearly labeled, appropriate containers, or managed as otherwise specified in the Work Plan (or equivalent), FSP, and/or IDW management guidance document.

Investigative-Derived Waste (IDW) generated during drilling activities, including soil and excess drilling fluids (if used), and decontamination liquids, will be stored on site in appropriately labeled containers and disposed of properly. Disposable materials will be stored and disposed of separately. Containers must be labeled at the time of collection and will include date, location(s), site name, city, state, and description of matrix contained (e.g., soil, PPE).

Waste will be managed in accordance with the *TGI for Investigation-Derived Waste Handling and Storage,* the procedures identified in the FIP/work plan or QAPP as well as state-, federal- or client-specific requirements. Be certain that waste containers are properly labeled and documented in the field log.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

If not using FieldNow®, all well drilling/installations activities will be documented on appropriate field/log forms as well as in a proper field notebook and/or Personal Digital Assistant (PDA) and/or tablet. All field data will be recorded digitally or with indelible ink. Field forms, logs/notes (including daily field and calibration logs), digital records, and chain-of-custody records will be maintained by the field team lead. Any deviations or omissions from this TGI will be documented.

Additionally, all documents (and photographs) should be scanned and electronically filed in the appropriate project directory for easy access. Pertinent information will include personnel present on site, times of arrival and departure, significant weather conditions, timing of well installation activities, soil descriptions, well construction specifications (screen and riser material and diameter, sump length, screen length and slot size, riser length, filter pack type and volume, type of well seal (fine sand or bentonite seal) and volume, type and volume of grout (neat cement or cement-bentonite), and other materials used.

Management of the original documents from the field will be completed in accordance with the site-specific QAPP. Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements.

Initial field logs and forms will be transmitted to the Arcadis CPM and/or Technical Lead at the end of each day unless otherwise directed by the CPM. The field team leader retains copies of the field documentation.

Locations of newly installed wells will be documented photographically and/or on a site sketch. If appropriate, a measuring wheel, engineer's tape, or handheld GPS will be used to determine approximate distances from key site features or estimated coordinates.

The well location, ground surface elevation, and inner and outer casing elevations will be surveyed using the method specified in the FIP/Work Plan. Generally, a local baseline control will be set up. This local baseline control can then be tied into the appropriate vertical and horizontal datum, such as the National Geodetic Vertical Datum (NGVD)of 1929 or North American Vertical Datum (NAVD) of 1988 and the State Plane Coordinate System. At a minimum, the elevation of the top of the inner casing used for water-level measurements should be measured to the nearest 0.01 foot. Elevations will be established in relation to the NGVD of 1929 or the NAVD of 1988. A permanent mark will be placed on top of the inner casing to mark the point for water-level measurements.

11 Quality Assurance

Quality assurance procedures will be conducted in accordance with the Arcadis Quality Management System or the site-specific QAPP. Refer to the QAPP or FIP/sampling plan/work plan for specific requirements.

All drilling equipment and associated tools (including augers, drill rods, sampling equipment, wrenches, and any other equipment or tools) that may have come in contact with soil will be cleaned in accordance with the procedures outlined in the appropriate TGI. All well construction materials will be inspected and cleaned (as necessary) prior to well installation.

Field-derived quality assurance blanks will be collected as specified in the FIP/work plan and/or site- specific QAPP, depending on the project quality objectives. Typically, field rinse blanks (equipment blanks) will be collected when non-dedicated equipment (e.g., split-spoon sampler, stainless steel spoon) is used during soil sampling. Field rinse blanks will be used to confirm that decontamination procedures are sufficient and samples are representative of site conditions. Trip blanks for VOCs, which aid in the detection of contaminants from other media, sources, or the container itself, will be kept with the coolers and the sample containers throughout the sampling activities and during transport to the laboratory.

Operate all monitoring instrumentation in accordance with manufacturer's instructions and calibration procedures. Calibrate instruments at the beginning of each day and verify the calibration at the end of each day. Record all calibration activities in the field notebook.

12 References

American Society for Testing Materials (ASTM) D5092 - *Standard Practice for Design and Installation of Ground Water Monitoring Wells*. American Society for Testing Materials. West Conshohocken, Pennsylvania.

American Society of Testing and Materials (ASTM) D1586 - *Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils*. American Society for Testing Materials. West Conshohocken, Pennsylvania.

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TGI – Vertical Aquifer Profile (VAP) Sampling

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Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
	0	June 22, 2018	All	Original SOP	Joe Quinnan
	1	May 11, 2020	Multiple	Added content to Personnel Qualifications, references to Geoprobe SP-16 and SP-22, Attachment 3, and updated references /formatting	Marc Killingstad
	2	June 15, 2022	Multiple	Combined with PFAS-specific VAP TGI and dye/tracer procedures.	Patrick Curry

Approval Signatures

Prepared by:		6/15/2022
	Patrick Curry (Preparer)	Date
Reviewed by:		6/15/2022
	Marc Killingstad (Subject Matter Expert)	Date

1 Introduction

This Arcadis Technical Guidance Instruction (TGI) describes proper vertical aquifer profile (VAP) sampling procedures using a variety of methods and approaches. This document has been developed to emphasize drilling and sampling procedures used to collect groundwater samples from boreholes installed via direct push technology (DPT), hollow stem auger (HSA), and rotary-sonic (sonic) methods and includes the use of visible tracer in drilling fluid to obtain representative samples during vertical profiling.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

Vertical aquifer profile (VAP) borings are typically advanced via DPT, HSA, or sonic drilling techniques to collect single or multiple depth-discrete groundwater samples using low-flow or grab sampling methodologies. This can be combined with retrieval of continuous soil cores and lithologic logging, as well as collection of single or multiple depth-discrete dry and saturated soil samples.

When possible, co-locate or bias VAP groundwater sampling intervals towards potential discrete transport zones (and target slow advection zones when feasible) as indicated by soil logging observations or permeability measurements (e.g., point slug tests, Geoprobe® hydraulic profiling tool [HPT] [preferred],

Waterloo APS™ [alternate]). Permeability measurements coupled with contaminant concentration allows estimationof relative flux and mass discharge to evaluate potential risk to downgradient receptors. In the absence ofpermeability measurements, field soil lithological logging observations may be used to interpret hydrostratigraphy and select sampling intervals.

The intent of this TGI is to provide VAP instructions including specific considerations for per- and polyfluoroalkyl substances (PFAS) due to their unique chemical and physical properties, low detection limits, and regulatory standards. It also covers field procedures for using nontoxic fluorescent tracer (e.g., fluorescein dye) in drilling fluid during drilling to assist in determining when sufficient purging has been performed prior to collecting screening-level groundwater samples during the drilling process. Screening level groundwater samples may be obtained by evacuating water from the drill casing or from intervals of geologic formations isolated by inflatable packers. This procedure improves the quality of screening-level groundwater samples by providing a technical basis to determine when purging has sufficiently removed drill water prior to collecting screening-level groundwater samples.

Multiple VAP samples can be collected through temporary wells, drilling rod tooling (e.g., Geoprobe® Screen Point 16 [SP-16]/Screen Point 22 [SP-22] Groundwater Samplers or SP-60 Sonic Groundwater Sampler or Cascade's Sonic Push-Ahead or Packer Isolation Groundwater Profilers) or via combined hydraulic profiling and sampling tools (e.g., Geoprobe® HPT-Groundwater Sampling System [HPT-GWS] or Waterloo APS™). They can be analyzed quickly via on-site mobile lab or expedited off-site fixed lab analysis to provide adaptive high-resolution quantitative groundwater concentration data. The vertical frequency of groundwater sampling within a formation will be determined relative to the scale of variability demonstrated in site hydrostratigraphy and outlined in the FIP/Work Plan. Thin aquifers with transport zones only tens of feet thick (or less) can be sampled at intervals as close as 3 to 5 feet. In aquifers with transport zones substantially thicker (e.g., more than 50 feet), sample spacing of 5 to 20 feet may be adequate. It is important to note that field data be evaluated to verify that sampling intervals provide sufficient resolution to meet data quality objectives (DQOs) (See **Section 7**).

4 Personnel Qualifications

In general, VAP activities will be performed by persons who have been trained in proper drilling and sampling procedures under the guidance of an experienced field geologist, engineer, or technician. Arcadis personnel overseeing, directing, or supervising VAP activities shall have previous related experience (minimum of 2 years) in drilling and groundwater sampling. Drilling subcontractors will need current applicable drilling licenses.

Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilization, the field team will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP)/field sampling plan, Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Additionally, the field team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

5 Equipment List

The following equipment and materials must be available for VAP sampling

- Site plan with proposed sampling locations
- Relevant work plan (or equivalent)
- Health and Safety Plan (HASP)
- Field Tablet with appropriate Fulcrum app for logging groundwater sampling.
- NOTE: *As of June 2022, several digital logging applications are available through the FieldNow™ program and the Fulcrum app. A future revision of this TGI, likely in early 2023, will emphasize digital logging methods and groundwater sampling. FieldNow™ is discussed further in Section 10.*
- Appropriate health and safety equipment, as specified in the site HASP
    - Drilling Equipment
    - Drill rig (to be provided by drilling subcontractor). Type (e.g., roto-sonic, direct push) to be determined based on site-specific details
    - Traffic cones, delineators, caution tape, and/or fencing as appropriate for securing the work area,if not provided by the drillers

      NOTE: *Prior to mobilizing to the site, Arcadis personnel will contact the drilling subcontractor or in-house driller (as appropriate) to confirm that appropriate sampling equipment will be provided in quantities capable of achieving estimated target depths. Typical equipment/materials providedby the driller could include*

        - Acetate or plastic liners
        - Appropriate length of drilling rods and tooling
        - Drilling and sampling equipment decontamination materials,
        - Decontamination pad materials, if required. See **Section 6.3** below for more information
- Sampling Equipment
    - Appropriate groundwater sampling equipment (e.g., disposable bailers for volumetric sampling,peristaltic pump for shallow groundwater sampling, submersible bladder pump for deeper sampling). Refer to *the TGI – Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells* (Arcadis 2020a) for necessary equipment
    - Direct push groundwater samplers (e.g., Geoprobe® SP-22) or roto-sonic sampling devices (e.g.,Cascade Push Ahead/Packer Isolation Groundwater Profiler or Geoprobe® SP-60 Sonic Groundwater Sampler) (to be provided by drilling subcontractor)
    - Appropriate soil sampling equipment (e.g., stainless steel spatulas, knife, metal trowel)

- Dedicated plastic sheeting (preferably high-density polyethylene [HDPE]) or other clean surface toprevent sample contact with the ground
- Multi-parameter water quality probe (e.g., conductivity, temperature, pH, dissolved oxygen,oxidation reduction potential, and turbidity meter)
- Water level meter
- Appropriate sample containers and labels
  - Labeled sample bottles: see the *TGI – Poly- and Perfluorinated Alkyl Substances (PFAS)Field Sampling Guidance* (Arcadis 2017a) for PFAS-specific considerations
  - Ziplock-style bags to hold ice and samples
  - Appropriate blanks (field reagent blanks supplied by the laboratory)
  - Packing and shipping materials
  - Chain-of-Custody (COC) Forms; see the *Sample Chain of Custody* for reference (Arcadis2017b)
  - Appropriate transport bottles (coolers) with ice and appropriate labeling, no blue ice

- Decontamination Equipment
  - Equipment cleaning materials: see the *TGI – Poly- and Perfluorinated Alkyl Substances (PFAS)Field Sampling Guidance* (Arcadis 2017a) or the *TGI – Groundwater and Soil Sampling Equipment Decontamination* (Arcadis 2020b) as applicable
  - Drum labels as required for investigation derived waste handling: see the *TGI – Investigation-Derived Waste Handling and Storage* for details (Arcadis 2017c)
- Documentation/Field Notes
  - Electronic data collection device (smart phone or tablet) as applicable
  - Pens, pencils, and/or Sharpie® brand pens for writing
  - Digital camera
  - Any other appropriate field forms
- Tracer Equipment (as needed)
  - Sodium fluorescein (also known as fluorescein or uranine dye) tracer (to be added to drilling water to produce a vibrant yellow-green color); 38 grams of dye will be added to each 500 gallons of drilling water (potable water) to achieve target applied tracer concentration of 20 milligrams per liter (mg/L)
  - Bottles for retaining dyed drilling water samples and preparing visual dye standards (clear, colorless, 40 mL unpreserved VOA bottles or equivalent)
  - Graduated cylinders (50 mL and 1 L)
  - Scale for measuring mass of dye to the nearest 1 gram
  - Bottles for groundwater samples to be analyzed for tracer dye (if necessary) and chemicals of concern (COCs)

- Poly storage tank (typically 550-to-1,000-gallon capacity)
- Potable water source
- Generator
- Utility pump for mixing dye
- Pump for groundwater purging and sampling
- Flashlight or other portable lighting device
- Blue ice (for tracer dye samples)

6 Cautions

Field activities associated with borehole advancement and VAP groundwater sampling will be performed in accordance with the HASP, a copy of which will be present on site during such activities. Field staff (including subcontractors) will be aware of and understand the associated physical and health hazards.

6.1 Utility Clearance

The appropriate drilling authorities will be contacted and a site visit for public utility line clearance at the proposed boring locations will be conducted at least 72 hours prior to work commencing. As applicable, utility maps will be reviewed during field reconnaissance of the proposed investigation locations to determine if any are co-located with public utility lines. Arcadis will also contract an independent geophysical survey company to verify that proposed boring locations are not co-located with existing underground utility/substructure features, as necessary. Arcadis will clear locations with soft dig methods to assess the presence of underground utilities as necessary.

See the *Utility Location and Clearance Arcadis Health and Safety Standard* (Arcadis 2020c) for reference.

6.2 PFAS-Specific General Sampling Considerations

This section provides a summary of methods and procedures applicable to the collection of environmental samples for field screening or laboratory analysis during PFAS site characterization activities. In general, sampling techniques used for PFAS site characterization are consistent with conventional sampling techniques used in the environmental industry, but special consideration is made regarding PFAS-containing materials and cross-contamination potential. For example, Teflon™ and other fluoropolymer containing materials are found in pumps, tubing, and sample storage containers and, therefore, will be avoided if possible (Department of Environment Regulation [DER], Western Australia 2016; New Hampshire Department of Environmental Services [NHDES] 2016). Certain field documentation materials such as waterproof paper or field books, adhesive paper products, and some writing utensils (grouped as non-Sharpie® markers) are also prohibited items during PFAS sampling (DER 2016; NHDES 2016).

Attachment 1 (Tables 1 and 2) provides recommendations for PFAS Site Investigation equipment. Table 1 provides a summary of materials that have been approved for site investigation; this list is expected to grow longer as industry experience increases. Table 2 provides a summary of field equipment and materials that have available testing information and/or industry knowledge regarding PFAS cross-

contamination potential, and it is recommended that these materials be prohibited for samplecollection. For materials that are suspected of containing and/or retaining PFAS, these recommendations are considered preliminary and subject to change.

Given the extremely low detection limits associated with PFAS analysis and the many potential sources of trace levels of PFAS, field personnel are typically advised to err on the side of caution by strictly following field wear guidelines and decontamination procedures as specified in the *TGI - Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance* (Arcadis 2022).The most important consideration during PFAS related VAP sampling is to prevent contact between sample media and suspect PFAS sources.

6.3 PFAS-Specific Groundwater Sampling

The potential presence of material containing PFAS in equipment that may come into contact with the target water sample must be evaluated as part of the sample planning process to maintain sample integrity. For example, low-flow sampling with a peristaltic pump will be conducted using silicone or HDPE tubing; Teflon™ tubing is prohibited (DER 2016). If a bladder pump is used to collect samples, the bladder and other internal parts (e.g., check balls, O-rings, compression fittings) will not contain Teflon™, and bladder and O-rings will be changed between samples (DER 2016).

Note that if high concentrations of PFAS related to Class B firefighting foams are expected in a groundwater sample, it has been recommended to collect and shake a small portion of the sample at the time of sample collection (USACE 2016; Arcadis 2017a). If foaming is noted within the sample, it indicates elevated concentrations of PFAS may be present and the sample will be proactively diluted at the laboratory prior to analysis, and the foaming will be noted on the sample COC form. It is recommended to collect sampling equipment blanks following foam observation to confirm the effectiveness of decontamination procedures.

6.4 Use of Tracer in Drilling Fluid

Field staff (including subcontractors) will be aware of tracer hazards and understand the associated health hazards. Please be sure to read the SDS (included as Attachment 2) for fluorescein dye. Note that some individuals can experience a mild allergic reaction to skin contact with fluorescein. Protective gloves will be worn during dye handling and mixing activities, and rinse bottles will be readily available for washing affected areas in case of accidental contact.

7 Health and Safety Considerations

To ensure the safety of the field personnel, field activities associated with VAP will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities. Review all site-specific and procedural hazards as they are provided in the HASP and review relevant Job SafetyAnalysis (JSA) documents in the field each day prior to beginning work.

Appropriate personal protective equipment (PPE) will be always worn in line with the task and the site-specific HASP. Verify staff has required health and safety training and personal protection

equipment in accordance with the HASP and JSAs. At a minimum, all staff will have level D PPE with chemical resistant gloves.

8 Procedure

The specific procedure for advancing VAP borings will be developed after careful review and consideration of project DQOs and clearly detailed in the FIP/Work Plan. Typically, VAP borings are conducted in boreholes adjacent to soil borings previously completed to develop stratigraphic and relative permeability profiles of the aquifer to determine sampling depth intervals that target transport zones. Prior boreholes typically consist of soil borings with detailed soil descriptions or Geoprobe® HPT borings. The primary advantage of completing stratigraphic/permeability profiles in advance of VAP sampling is to gain an understanding of hydrofacies trends to ensure that the most appropriate intervals and sampling methods are used. For sonic or HSA drilling, VAP samples are typically collected from the same borehole as soil samples. In the absence of a co-located boring, sample depth intervals can be determined based upon lithologic logging of soil cores, either from a separate adjacent borehole or from the same borehole. HPT drilling will be completed consistent with the *TGI – Geoprobe Hydraulic Profiling Tool (HPT)* (Arcadis 2022a), and soil lithologic logging will be performed in accordance with *TGI – Soil Description* (Arcadis 2022b).

NOTE: *Waterloo APS™ can be utilized as an alternative to HPT to provide permeability profiles, but it is more time consuming than HPT; therefore, it is not considered the preferred tool for permeability profiling.*

8.1 Direct Push Vertical Aquifer Profile Sampling

Direct push tooling is ideal for shallow unconsolidated aquifers and requires minimal water for drilling, reducing the potential for sample dilution/cross-contamination. For sites with shallow groundwater in unconsolidated formations (e.g., at less than 100 feet bgs), the typical approach is to collect VAP groundwater samples nominally every 5 to 10 feet with a bias to the more permeable transport zones.

When a zone of interest is identified, either by using permeability measurements (preferred) or logging soil, a screen point sampling device such as Geoprobe® SP-16 or SP-22 (see **Attachment 3**) can be driven to the target interval and the screen opened to collect a groundwater sample. In poorly sorted aquifers with appreciable amounts of silt, VAP sampling from an adjacent borehole after completing initial permeability profiling (e.g., Geoprobe® HPT or point slug tests) is typically more efficient and cost effective. In the absence of permeability profiling tools (e.g., HPT), VAP sampling can be performed based on soil lithological observations alone, either from a separate borehole or in the same borehole. However, VAP sample collection can be more efficient when conducted from an adjacent borehole, particularly if a bottom-up sampling approach is used. See Section 8.1.1.

Combined permeability profiling and sampling tools such as the Geoprobe® HPT-GWS (or Waterloo APS™ as an alternate) can be used to collect groundwater samples based on permeability measurements from the same borehole at deeper depths where the process is more cost-effective; otherwise at shallower depths, separate permeability profiling prior to VAP sampling is preferred. This is most effective in well-sorted sand and gravel when small volumes are required for analysis, since these tools provide limited volumes for purging and sample collection. Use of these combined tools (HPT-GWS or Waterloo APS™) for PFAS sites is not recommended because low detection and regulatory thresholds for PFASs require more extensive purging to decontaminate the sampling equipment (i.e., insufficient data are available to confirm the volume of purging required to eliminate cross-contamination with PFAS).

It is recommended that DPT drilling for VAP sampling be completed using a dual-tube drilling approach. An outer casing is advanced with the screen point sampling device to limit the potential for cross-contamination between sampling intervals. Pre-calculated volume purging and monitoring for water quality parameter stabilization can be performed consistent with low-flow sampling to help determine when purge water is representative of the groundwater formation.

There are two general methods for completing VAP sampling: bottom-up and top-down. With bottom-up sampling, a greater purge volume is required to ensure a representative groundwater sample; however, the overall time savings is significant relative to top-down sampling, where more time is required per borehole to lower the tooling, retract it, and decontaminate it between subsequent sampling intervals. However, the top-down method minimizes any potential for cross-contamination and is the preferred approach for PFAS projects due to the low detection limits and regulatory levels associated with PFASs.

8.1.1 Bottom-Up VAP Sampling

Bottom-up VAP sampling involves advancing dual-tube direct push casing to the deepest target depth with either a solid drive tip (without collecting soil cores) or acetate liners for collection of continuous soil cores to provide a lithological log for the entire boring. This is followed by lowering the groundwater sampling screen through the outer casing to the bottom of the borehole and collecting multiple VAP groundwater samples at different depths as the casing and screen is retracted up the borehole.

Using this approach, the external casing is retracted to allow borehole collapse around the sampling screen while isolating it from the section above that is still covered by the external casing.

NOTE: *Bottom-up VAP sampling is not recommended when there is a potential for dense non-aqueous phase liquid (DNAPL), the highest concentrations are expected to be at the bottom of the formation, or the borehole goes through multiple confining units. Bottom-up is not recommended for PFAS sampling due to potential cross-contamination concerns associated with lack of decontamination between sample intervals.*

The advantages of this approach are: (1) when combined with soil core collection, groundwater sampling depth intervals can be pre-selected based on lithologic observations to target the transport zones, especially in the absence of any co-located permeability measurements, and (2) the entire process is much more time-efficient per borehole as the sampling equipment is not pulled, decontaminated, and then drilled to the next interval. However, additional purging (i.e., 3to 5 casing volumes) is required to assure a representative groundwater sample. Bottom-up sampling also does not allow for post-grouting of the borehole since when the groundwater sampling device is pulled up to the next VAP sampling interval, the sidewalls of the open borehole below collapse.

8.1.2 Top-Down VAP Sampling

Top-down VAP sampling involves advancing dual-tube direct push casing with either a solid drive tip (without collecting soil cores) or a plastic liner for soil core retrieval from target depth interval followed by lowering the screen point sampling screen to target depth and pulling up the outer casing to expose the screen. After purging and sample collection, the screen point device is pulled back up and decontaminated. A solid drive tip or plastic liner is lowered back into the borehole and the entire assembly is then advanced to the next depth interval. Thus, top-down sampling requires pulling the tooling after each sample interval, decontaminating the tooling, re-setting the groundwater sampler, and advancing the tooling to the next planned interval.

The advantages of this approach are that it allows grouting of the borehole from the bottom of the borehole and

reduces the potential for cross-contamination from adjacent sampling intervals.

The primary disadvantage is that the entire process is much less time-efficient per borehole since the tooling must be retracted and re-advanced every time.

NOTE: *Top-down is the preferred method for PFAS VAP sampling.*

8.2 Sonic Drilling VAP Sampling

For sites with deep unconsolidated aquifers, bedrock/weathered bedrock, or otherwise challenging drilling conditions (e.g., presence of dense tills, caliche, cobbles), sonic drilling will be necessary to complete VAP. Like direct push, groundwater profilers can be used with sonic rigs to collect multiple depth-discrete groundwater samples biased towards transport zones based on soil lithological cores. The configuration of individual samplers varies based on their manufacturer by different drilling contractors (e.g., Cascade Push Ahead/Packer Isolation Groundwater Profiler or Geoprobe® SP-60).

The overall strategy of sonic drilling VAP sampling is consistent with direct push VAP sampling; however, drilling with sonic or some rotary methods can require the introduction of drilling fluid/water that can potentially affect the integrity of the groundwater sample. If possible, sonic drilling for VAP borings will be conducted without the use of drilling water. If, for example, the geology is known for flowing sands or VAP is required deep below the water table, drilling water will be used to keep the core barrel free inside the outer drill casing. In this case it is recommended that a visible dye be used to spike the drilling water to assist with purging of the VAP interval. The VAP interval is then purged until the visible dye is no longer visible, or less the 10% of the starting concentration. For more on drilling with visible dye, see Section 8.2.3. Sonic VAP sampling is typically performed in a top-down manner using either a push ahead sampling device or a packer system.

8.2.1 Push-Ahead Groundwater Profiler

Push-ahead groundwater sampling devices are available through Cascade Drilling and other sonic drillers and consists of a stainless-steel sheathed “screen” threaded to the base of the sonic drill rod. The device is driven ahead of the sonic casing into the undisturbed formation to the prescribed sample depth interval. Once the point is at the specified interval, the threaded portion between the profiler and rod is partially unthreaded to expose the water ports to allow native formation water to enter the profiler. A groundwater sample is then obtained using either a stainless-steel bailer or pump with tubing depending on the DQOs. The interval is typically purged until relatively free of fine-grained material.

The disadvantage of using this device is that groundwater samples are obtained from undisturbed native formation with unknown soil lithology, so sampling is conducted “blind”. Therefore, it is recommended that a pre-existing lithological log from an adjacent borehole is used to determine sampling depth intervals.

8.2.2 Packer Isolation Groundwater Profiler

Packer Isolation groundwater profilers (e.g., Packer Isolation groundwater profiler from Cascade, Geoprobe ® SP-60 Sonic Groundwater Profiler) work by driving the casing over the soil core, retrieving the soil core barrel, and then lowering a stainless screen and packer assembly to the base of the sonic casing. The sonic outer casing is then extracted to expose the screen to the formation, and the packer is inflated within the casing above the screen to isolate the screened interval from any water that might be above the packer in the sonic casing. A groundwater sample can then be collected from the screen.

The biggest advantage of this device is that groundwater sampling depth intervals can be determined based on lithological logs obtained from the same borehole.

A disadvantage is that a large volume of purge water may need to be removed to clear the borehole of water introduced from above.

8.2.3 Drilling with Visible Dye

Potable water is commonly used as a drilling fluid during drilling to remove cuttings of geologic materials from the borehole (e.g., coring or roller-bit rotary drilling), cool the drill bit (e.g., sonic drilling), and/or maintain sufficient hydraulic pressure within the drilling tools to prevent heaving of aquifer solids into the drill casing(s).

Typically, if groundwater sampling is performed during drilling, the purge volume to be removed is at least as much as was lost during drilling. However, accurately determining the volume of water lost to the formation, or to specific intervals of the borehole, is not always feasible or possible.

To ensure that groundwater samples accurately represent groundwater quality of the surrounding formation and are not significantly influenced by unrecovered drilling fluid, fluorescein dye can be added to the drilling water to visually confirm when unimpacted native groundwater enters the borehole and can be sampled.

The target concentration of dye is approximately 20 mg/L, which is greater than two (2) orders of magnitude above its visual threshold (approximately 0.1 mg/L) and over five (5) orders of magnitude above its typical laboratory detection limit (less than 0.001 mg/L). Once the drilling tool has been advanced to the prescribed depth for groundwater sampling, water will be pumped from the borehole until the discharge water is relatively clear of fluorescein. The goal of purging is to reach the clarity of a prepared visual standard, indicating that the discharge water is comprised of at least 95 percent formation water and less than 5 percent drilling water. Groundwater samples will then be collected for COC analysis.

If the visual standard is still not reached after a reasonable period and volume of purging, then COC sampling can still be performed, provided that samples of the dyed drilling water and groundwater are also sent for fluorescein analysis. The fluorescein data can then be used to calculate a correction factor to be applied to COC analytical results.

8.2.3.1 Set-Up Procedures

a. Dye Batch Preparation

- Prior to initiating drilling activities, measure the proper mass of powdered dye for mixing with drilling water - 38 grams of fluorescein (provided by Ozark Underground Laboratory) will be added to every 500 gallons of water to yield an average tracer concentration of approximately 20 mg/L.
- If the drilling water “batch” is larger or smaller than 500 gallons, the same ratio of dye to drilling water will be used.
- Measure the mass of dye using a scale with an accuracy of +/- 1 gram.
- Add the dye to the drilling water batch tank while also adding the potable water to provide agitation to assist in mixing the dye.
- A utility pump is also recommended to mix the tracer with the drilling water by recirculating water in the tank for at least 15 minutes.

- Place 40 mL of the dyed drilling water into a 50 mL graduated cylinder for use in preparing the visual standard discussed below.
- Collect four (4) additional 40 ml unpreserved VOA vials of drilling water from each batch of drilling water – label all four of these vials "DW1" for the first batch of drilling water, "DW2" for the second batch, etc. These samples will be archived for potential use in preparing other standards with other dilutions (optional) or for submittal for laboratory analysis, if necessary.

b. Preparation of Visual Standard: A visual standard will be prepared for each batch of dyed drilling water, as follows.

- Pour the 40 mL volume of dyed drilling water from the 50 mL graduated cylinder into a 1 L graduated cylinder.
- Add 760 mL of un-dyed potable water (from the same potable water source used to prepare the dyed drilling water) to the 1 L graduated cylinder to produce 800 mL of "visual standard".
- Fill one 40-mL unpreserved VOA vial with visual standard solution and label this "VS1" for the visual standard from the first batch of drilling water, "VS2" for the visual standard from the second batch of drilling water, etc.
- These visual standards represent a 95% dilution of the drill water and will provide a visual standard to verify that sufficient purging has been performed to remove at least 95% of the drilling water from a given interval, indicating that the purge water consists of at least 95% formation water.
- Discard the remaining fluid within the graduated cylinder using an appropriate container.
- Photograph the "DW" samples and the "VS" sample from each batch of drilling water with adequate, consistent light, against a white background.
- Keep all the dyed drilling water ("DW") samples and visual standard ("VS") samples in a cooler to keep them dark as the dye will degrade with exposure to light.

8.2.3.2 Drilling Procedures

- Fresh drilling water from the dyed drilling water batch tank will be used during drilling operations. In general, a positive head will be maintained during drilling, which should prevent dilution of the drilling water by formation water. However, any water upwelling from the casing during drilling will be contained in a tub positioned over the borehole. As needed, recovered water in the tub will be pumped to a frac tank.
- The drilling water source will be sampled for chlorine and pH at the start of the project. Chlorine, if present in detectable quantities, will consume fluorescein; therefore, wait a period of at least four (4) hours between dye addition and sampling (and use) of the drilling fluid. Below pH values of about 5, fluorescein will have reduced fluorescence. Depending on the source of the drilling fluid and project objectives, the source water may also be sampled for COCs and fluorescein.
- In open sunlight, fluorescein photodegrades rapidly. If the tracer batch tank is translucent, use 1-millimeter (mm) thick black plastic to cover the tank during the day to minimize photodegradation of the tracer batch water.
- After tracer addition, each batch of drilling fluid will be sampled at least once for fluorescein.
- At the end of the day, any excess tracer batch water can be stored for use on the following day, or it may be disposed of as investigation derived waste. Alternatively, fluorescein concentrations can be reduced to below

visible concentrations with granular activated carbon, UV-oxidation, chemical oxidants, or direct exposure to sunlight for several days.

- The field geologist will record the amount of drilling water lost to the formation during drilling of each sampling depth interval.
- At the end of the project, any excess tracer batch water can be disposed of as investigation derived waste. Alternatively, fluorescein concentrations can be reduced to below visible concentrations with granular activated carbon, UV-oxidation, chemical oxidants, or direct exposure to sunlight for several days. Depending on project and regulatory requirements, excess batch water with fluorescein concentrations below the visible limit could be discharged to a sanitary sewer or other discharge location.

8.2.3.3 Purging and Sampling Procedures

- After a groundwater sampling interval is reached, purging and screening-level groundwater sampling will be performed.
- The target sample interval will be purged using a pump, and during purging, purge water will be periodically collected in a 40-mL unpreserved VOA vial and compared to the visual standard ("VS" sample) prepared from the drilling water used to drill that depth interval.
- If the purge water contains significant suspended particulates/turbidity, it may be necessary to allow particulates to settle before comparing the purge water sample to the visual standard.
- Purging will continue until ***one*** of the following two conditions is met:

    1) Purge water clarity (in terms of remaining dye content) matches or exceeds the clarity of the visual standard, indicating that the purge water consists of at least 95% formation water.

        a. In this case, the purge water sample and the associated visual standard will be photographed against a white background to document that the purging goal has been reached.

OR

    2) A different practical purging limit has been reached, based on purge volume or time

        a. In this case, the purge water sample and the associated visual standard will be photographed against a white background to document the degree of purge water visual clarity that was attained

        b. Also, a sample of the purge water will be collected in a 40 mL unpreserved VOA; this sample and one of the vials of dyed drilling water will be submitted to Ozark Underground Laboratories for quantitative analysis of fluorescein. These samples will be shipped in a cooler with reusable "Blue Ice" rather than wet ice. The analytical results for fluorescein will be used to calculate a COC correction factor, as discussed below (see Section 8.2.3.4).

- After purging has been completed, screening-level groundwater samples will be collected from the discharge end of the pump tubing for COC analysis in accordance with the approved work plan.

8.2.3.4 Calculation of Correction Factor

- If the purge water does not reach the goal indicated by the visual standard ("VS" sample), a sample of the drilling water and a sample of the purge water (obtained immediately prior to sampling for COC analysis) will be sent for laboratory analysis of fluorescein.
- Representative COC concentrations in groundwater (C_{gw}) can then be calculated as:

$$C_{gw} = C_m [F_d / (F_d – F_s)]$$

where: C_m = measured COC concentration, as reported by the lab

F_d = fluorescein concentration in drilling water

F_s = fluorescein concentration in groundwater sample

- The term $[F_d / (F_d – F_s)]$ is the COC correction factor.

8.3 Boring Abandonment

Upon completion, each top-down borehole is backfilled with bentonite grout from the terminal end of the boring upward. The top portion of each boring is sealed with asphalt or concrete to match the existing grade. Each bottom-up borehole is typically abandoned by the collapse as the rods are retraced.

Borehole abandonment requirements in some geographies dictate top-down sampling; this should be verified in advance of work and outlined in the FIP/Work Plan. See also *TGI for Monitoring Well and Borehole Decommissioning.*

9 Waste Management

Investigation derived waste (IDW) (e.g., soil cuttings and decontamination water generated during cleaning procedures) will be collected and contained on site in appropriate containers: see the *TGI – Investigation- Derived Waste Handling and Storage* for details (Arcadis 2020d). All IDW generated during field activities will be placed in Department of Transportation approved containers, sealed, and labeled. Containerized IDW will be stored on site until it is profiled and subsequently transported to an approved facility for disposal or recycling.

Personal protective equipment (e.g., gloves, disposable clothing, disposable equipment) resulting from personnel cleaning procedures and soil sampling activities will be placed in plastic bags. These bags will be transferred into appropriately labeled containers for appropriate disposal.

Waste manifests for all IDW suspected to have come into contact with PFAS will clearly note the potential presence of PFAS.

Additional IDW sampling and management details will be provided in the site-specific FIP/Work Plan/QAPP addendum and will be consistent with applicable client and state requirements.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

The supervising field lead will be responsible for documenting drilling events and for recording all relevant information in a clear and concise format. The record of drilling events will include (at a minimum):

- Start and finish drilling dates
- Project name and location
- Project number, client, and site location
- VAP boring number and depths
- Depth to water
- Type of VAP performed and associated tools
- Core barrel size
- Names of contractor's drillers, inspectors, or other people onsite
- Weather conditions

Field staff will ensure COC Forms are properly completed and will verify which analytes (including PFAS analytes) are required for analysis and note on the COC.

Waterproof field books must not be used for field notes. Instead, it's recommended that field notes be on loose paper on Masonite, plastic, or aluminum clip boards and/or electronic data collection tablets (as required). Other requirements for field notes include:

- Keep field notes, writing implements, and electronic data collection tablets away from samples and sampling materials; and,
- Do not write on sampling bottles unless they are closed.

11 Quality Assurance

In general, the following quality assurance and quality control (QA/QC) samples will be collected:

- Equipment blanks
- Field duplicates
- Field (i.e., reagent) blanks

- Matrix spike/matrix spike duplicate

Details on QC sampling requirements (e.g., frequency of collection, types of QA/QC samples) are provided in the QAPP and/or FIP/Work Plan. Additionally, detailed procedures related to equipment and field (i.e., reagent) blank sample collection are outlined in the *TGI for Equipment and Reagent Blank Sample Collection for PFAS Analysis*.

In general, equipment blanks should be collected from every piece of downhole equipment that could come into contact with soil or groundwater during sample collection. This includes the profiling tools (e.g., Geoprobe® SP-16, Geoprobe® SP-22, Geoprobe® SP-60, Cascade Packer Isolation Groundwater Profiler).

To avoid cross-contamination during drilling and sampling, reusable equipment such as hand tools will be cleaned using a non-phosphate soap free of VOCs, double-rinsed in potable water, and allowed to air dry prior to re-use. Drive casings and other drilling equipment will be steam cleaned or replaced with new equipment between boreholes. The drilling equipment will be cleaned in an area designated by the supervising engineer or geologist that is located outside of the work zone.

Prior to initiating field activities, water sources to be used during drilling activities will be sampled to verify those sources are PFAS-free to the extent possible.

Refer to quality control requirements for the project to ensure that appropriate quality assurance and quality control (QA/QC) samples are collected. When collecting QA/QC samples, the same guidelines apply as when collecting regular samples – specifically that:

- Samples will be collected in laboratory-supplied HDPE bottles
- Bottle caps must remain in the hand of the sampler until replaced on the bottle
- Labels must be completed after the caps have been placed back on each bottle
- Samples must be stored in appropriate transport bottles (coolers) with ice (Ziplock-type bags for use as ice containers) with appropriate labeling
- Do not use blue ice except for shipping fluorescein samples
- Store PFAS samples in a separate cooler from other types of samples

11.1 Equipment Blanks (if relevant)

QA/QC sampling typically includes daily collection of equipment blanks using the laboratory-supplied water, or in the case of PFAS sampling, “PFAS-free” water. For peristaltic pump tubing, laboratory supplied water will be poured into a clean HDPE sample bottle and then pumped through new HDPE tubing using the peristaltic pump (with new silicone tubing). Equipment blanks will also be collected from the water used by drillers, as well as any downhole tooling to ensure absence of any cross-contamination. Drilling water sources must be submitted for analysis of all COCs before work commences for VAP as discussed above. See *also TGI for Poly- and Perfluorinated Alkyl Substances (PFAS) Potable Water Sampling Guidance*.

11.2 Field Duplicates

QA/QC sampling typically includes the collection of one field duplicate for every 10 or 20 samples collected. Each duplicate sample will be collected immediately after the initial sample of which it is a duplicate into a separate laboratory-provided sample bottle. Do not indicate to the laboratory which sample the duplicate replicates (i.e., it will be given a blind reference on the COC and sample name such as “Dup 1”).

11.3 Field Blanks

QA/QC sampling for typically includes the submission of one laboratory supplied field blank per day. The reagent field blank sample is brought to the site in a laboratory-supplied sample bottle. Field staff transfer the laboratory-supplied reagent blank to an empty sample bottle. This reagent fieldblank will be placed in the same cooler as the other PFAS samples.

11.4 Matrix Spikes (optional in some cases)

QA/QC sampling includes submitting a sample to be used as a matrix spike if the project requires it. If a separate sample bottle is required, an additional sample will be collected immediately after the initial sample of which it is a duplicate into a separate laboratory-supplied sample bottle.

11.5 Laboratory Analytical QA/QC

- Internal laboratory QA/QC will consist of one laboratory blank and one laboratory control sample (or blank spike) per batch of samples, and additional QA/QCs as indicated by the laboratory QA/QC procedures. Isotope dilution will be used for quantification with isotope-labeled surrogate standards, as available.
- For groundwater and surface water samples, extract the entire groundwater and surface water sample and at least two sampling bottle solvent rinsates for analysis to increase sample accuracy. Avoid sub-sampling an aliquot of the sample bottle.
- Soil samples will be analyzed in their entirety or thoroughly homogenized before extraction and analysis.
- As part of the internal QA/QC, relative percent difference will be calculated between samples and corresponding field or laboratory duplicates. The laboratory quality assurance portion of the laboratory certificates will be reviewed to verify that all calculations/recoveries were within acceptable limits as established by the laboratory method.
- In January 2017, the U.S. Department of Defense and U.S. Department of Energy Quality Systems Manual (QSM) 5.1 (U.S. Department of Defense 2017) was finalized and introduced laboratory guidance for the measurement of PFAS in matrices other than drinking water. This guidance is not a detailed procedural method such as an U.S. Environmental Protection Agency method, but it does recommend best practices around the analysis of PFAS. Laboratories are not required to comply with QSM 5.1 until 2019, although the recommendations around PFAS analysis are similar to what most laboratories are already implementing. Arcadis recommends that any request for PFAS analysis in

groundwater or soil should specifically reference the need to comply with Table B-15 in the QSM 5.1 (i.e., Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water); however, this list can be modified to support project specific deliverables.

12 References

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ATTACHMENT 1

Table 1 and Table 2: PFAS Investigation Material Recommendations

Table 1: Summary of Acceptable Sampling Equipment and Materials for PFAS Site Investigations

Sampling Materials	Additional Considerations	References
Water Sampling Materials		
High density polyethylene (HDPE) or silicone tubing materials	--	DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
HDPE HydraSleeves™	Low density polyethylene (LDPE) HydraSleeves™ are not recommended	USACE 2016; MassDEP 2017
Drilling and Soil Sampling Materials		
PFAS-free drilling fluids	--	DER 2016
PFAS-free makeup water	Confirm PFAS-free water source via laboratory analysis prior to investigation	--
Acetate liners	For use in soil sampling	USACE 2016
Sample Containers and Storage		
HDPE sample containers with HDPE lined lids for soil and water samples	Laboratory should provide; whole bottle analysis of aqueous samples combined with a solvent rinse of bottle is recommended	DER 2016, MassDEP 2017
Ice contained in plastic (polyethylene) bags (double bagged)	--	DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Field Documentation		
Sharpie®	--	NHDES 2016; USACE 2016; MassDEP 2017
Ball point pens	--	MassDEP 2017
Standard paper and paper labels	--	DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Decontamination		
Water-only decontamination	Confirm PFAS-free water source via laboratory analysis prior to investigation	DER 2016
Alconox®, Liquinox® or Citranox® followed by deionized water or PFAS-free water rinse	Alconox® known to contain trace levels of 1,4-dioxane	NHDES 2016; USACE 2016; MassDEP 2017
Methanol, isopropanol, or acetone	Special health and safety precautions are necessary	UNEP 2015; USACE 2016

Note: This list is considered preliminary and additional materials may be added as additional information becomes available. Project teams are expected to follow a methodical evaluation process of materials to be used and confirm acceptance prior to implementation of field activities.

Table 2: Summary of Equipment and Materials Not Recommended for PFAS Site Investigations.

Sampling Materials	Known PFAS-Containing Materials	Suspected PFAS-Containing Materials	Materials with Potential to Retain PFASs	References
Water Sampling Materials				
Teflon® or polytetrafluoroethylene (PTFE)-containing or coated field equipment (e.g., tubing, bailers, tape, plumbing paste)	x			DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Passive diffusion bags			x	MassDEP 2017
LDPE HydraSleeves ™			x	USACE 2016; MassDEP 2017
Water particle filters			x	MassDEP 2017
Drilling and Soil Sampling Materials				
Aluminum foil			x	DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Drilling fluid containing PFASs	x	x		DER 2016
Sample Containers and Storage				
Glass sample containers with lined lids			x	DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
LDPE containers and lined lids			x	USACE 2016
Teflon® or PTFE- lined lids on containers (e.g., sample containers, rinsate water storage containers)	x			DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Reusable chemical or gel ice packs (e.g., BlueIce®)		x		DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Field Documentation				
Self-sticking notes and similar office products (e.g., 3M Post-it-notes)		x		DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Waterproof paper, notebooks, and labels	x			DER 2016, MassDEP 2017
Non-Sharpie® markers		x		NHDES 2016
Decontamination				
Some detergents and decontamination solutions (e.g., Decon 90® Decontamination Solution)	x	x		DER 2016; NHDES 2016; MassDEP 2017

N**ote**: For materials that are suspected of containing PFASs, or have the potential to retain PFASs, project specific considerations may provide adequate justification for use during the field event. For example, further evaluation may be conducted in the form of pre-field equipment blank sample analysis.

ATTACHMENT 2

Safety Data Sheet (SDS) Fluorescein

SAFETY DATA SHEET (SDS)
REVISION DATE: 03/03/2016

HUE CORPORATION

Color your everything, may your Hue come true

SECTION I. IDENTIFICATION OF THE SUBSTANCE/MIXTURE AND OF THE COMPANY/UNDERTAKING

PRODUCT IDENTIFIER:

PRODUCT NAME ..... **HUE URANINE CONC** (Also known as Fluorescein)
PRODUCT NUMBER 1-C8-073PC
COLOR INDEX NAME ..... ACID YELLOW 073
COLOR INDEX NO 45350
C. A. S. # 518-47-8
CHEMICAL FAMILY..... XANTHENE

INTENDED USE OF THE PRODUCT:

FELT TIP, MARKER INKS, WATER BASED COATINGS AND LEAK DETECTION

NAME, ADDRESS AND TELEPHONE OF RESPONSIBLE PARTY:

HUE CORPORATION	TELEPHONE	714-389-3130
P.O. BOX 509	FAX	714-389-9731
TUSTIN, CA 92781	EMAIL	SUPPORT@HUECORPORATION.COM

EMERGENCY TELEPHONE NUMBER:

CHEMTREC (USA)	1-800-424-9300
CHEMTREC (OUTSIDE USA)	1-703-527-3887

SECTION 2. HAZARD(S) IDENTIFICATION

CLASSIFICATION OF THE SUBSTANCE OR MIXTURE:

GHS-US
ACUTE TOX. - INHALATION (CATEGORY 5)
EYE DAM./IRRITATION (CATEGORY 2B)
SKIN CORR./IRRITATION (CATEGORY 3)

GHS LABELING:

HAZARD PICTOGRAMS (GHS-US):	NO SYMBOL
SIGNAL WORD	WARNING
HAZARD STATEMENT(S)	H333 - MAY BE HARMFUL IF INHALED H320 - CAUSES EYE IRRITATION H316 - CAUSES MILD SKIN IRRITATION
PRECAUTIONARY STATEMENTS	P305 + 351 + P338 - IF IN EYES: RINSE CAUTIOUSLY WITH WATER FOR SEVERAL MINUTES. REMOVE CONTACT LENSES IF PRESENT AND EASY

TO DO. CONTINUE RINSING.
P337 + P313 - IF EYE IRRITATION OCCURS/PERSISTS:
GET MEDICAL ADVICE AND ATTENTION.
P261 - AVOID BREATHING DUST/FUMES/GAS/MIST/VAPORS/SPRAY
P264 - WASH FACE THOROUGHLY AFTER HANDLING.
P322 + P313 - IF SKIN IRRITATION OCCURS: GET MEDICAL ADVICE/ ATTENTION.
P304 + 312 - IF INHALED: CALL A POISON CENTER/DOCTOR/PHYSICIAN IF YOU FEEL UNWELL

OTHER HAZARDS NO DATA AVAILABLE
UNKNOWN ACUTE TOXICITY NO DATA AVAILABLE

SECTION 3. COMPOSITION / INFORMATION ON INGREDIENTS

DESCRIPTION OF MIXTURE: PROPRIETARY MIXTURE OF DYES.

SUBSTANCE:

NAME	C.A.S.#	WEIGHT 100%	GHS-US CLASSIFICATION
ACID YELLOW 073	518-47-8	100%	ACUTE TOX. - INHALATION (CATEGORY 5) EYE DAM./IRRITATION (CATEGORY 2B) SKIN CORR./IRRITATION (CATEGORY 3)

SECTION 4. FIRST AID MEASURES

FIRST AID MEASURES GENERAL:

INHALATION: REMOVE TO FRESH AIR. IF BREATHING IS DIFFICULT, GIVE OXYGEN AND GET IMMEDIATE MEDICAL ATTENTION.

SKIN: WASH WITH MILD SOAP AND WATER. IF IRRITATION OCCURS GET MEDICAL ATTENTION. IF CLOTHING IS CONTAMINATED, RE-MOVE AND WASH BEFORE REUSE.

EYES: FLUSH EYES WITH WATER FOR AT LEAST 15 MINUTES, HOLDING EYELIDS APART FOR THOROUGH IRRIGATION. GET IMMEDIATE MEDICAL ATTENTION.

INGESTION: INDUCE VOMITING - SEEK IMMEDIATE MEDICAL ATTENTION.

MOST IMPORTANT SYMPTOMS AND EFFECTS, ACUTE AND DELAYED:

THIS PRODUCT IS NOT HAZARDOUS AS DEFINED BY HAZARDOUS COMMUNICATION STANDARD. HOWEVER, AS WITH ALL CHEMICAL; HANDLE WITH CARE, AVOID EYE AND SKIN CONTACT, AVOID INHALATION OF DUSTS OR VAPORS. WASH THOROUGHLY AFTER HANDLING. KEEP CONTAINERS CLOSED.

SECTION 5. FIRE-FIGHTING MEASURES

EXTINGUISHING MEDIA:

WATER, DRY CHEMICAL, CARBON DIOXIDE, FOAM.

SPECIAL HAZARDS ARISING FROM SUBSTANCE OR MEDIA:

FIREFIGHTERS SHOULD BE EQUIPPED WITH SELF-CONTAINED BREATHING APPARATUS TO GUARD AGAINST POTENTIALLY TOXIC AND IRRITATING FUMES. AVOID DUSTING. DUST CAN FORM EXPLOSIVE MIXTURES WITH AIR.

PROTECTION/ADVICE FOR FIREFIGHTER(S):

BE EQUIPPED WITH SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING.

SECTION 6. ACCIDENTAL RELEASE MEASURES

PERSONAL PRECAUTIONS:

REMOVE PERSONS FROM DANGER AREA.

ENVIROMENTAL PRECAUTIONS:

AVOID ANY UNCONTROLLED RELEASE OF MATERIAL. DO NOT EMPTY INTO DRAINS OR THE AQUATIC ENVIRONMENT.

EMERGENCY PROCEDURES:

NO ADDITIONAL INFORMATION

METHODS AND MATERIALS FOR CONTAMINENT AND CLEANING UP:

WHERE SPILLS ARE POSSIBLE, A COMPREHENSIVE SPILL RESPONSE PLAN SHOULD BE DEVELOPED AND IMPLEMENTED. AVOID ANY UNCONTROLLED RELEASE OF MATERIAL.

UTILIZE RECOMMENDED PROTECTIVE CLOTHING AND EQUIPMENT (SEE SECTION 8).
SPILLS SHOULD BE SWEPT UP USING AN ABSORBENT DUST CONTROL PRODUCT AND PLACED IN CONTAINERS. SPILL AREA CAN BE WASHED WITH WATER. COLLECT WATER FOR APPROVED DISPOSAL.
IN THE EVENT OF UNCONTROLLED RELEASE OF THIS MATERIAL, THE USER SHOULD DETERMINE IF THE RELEASE IS REPORTABLE UNDER APPLICABLE LAWS AND REGULATIONS.

SECTION 7. HANDLING AND STORAGE

PRECAUTIONS FOR SAFE HANDLING:

HANDLE WITH CARE. AVOID OVER EXPOSURE. USE NIOSH/OSHA APPROVED RESPIRATOR, WORK GLOVES, AND CLOTHING. WASH AFTER HANDLING. SENSITIVE INDIVIDUALS MAY EXPERIENCE RESPIRATORY ALLERGIES. MAY CAUSE SKIN IRRITATION. USE WITH LOCAL VENTILATION.

CONDITIONS FOR SAFE STORAGE, INCLUDING ANY INCOMPATIBILITIES:

USE PROCESS ENCLOSURES, LOCAL EXHAUST VENTILATION OR OTHER ENGINEERING CONTROLS TO KEEP AIRBORNE LEVELS BELOW RECOMMENDED EXPOSURE LIMITS.

KEEP AWAY FROM HEAT. KEEP AWAY FROM SOURCES OF IGNITION.

KEEP AWAY FROM STRONG OXIDIZING AND REDUSING AGENTS.

SPECIFIC END USES:

FELT TIP, MARKER INKS, WATER BASED COATINGS AND LEAK DETECTION

SECTION 8. EXPOSURE CONTROLS /PERSONAL PROTECTION

CONTROL PARAMETERS:

INGREDIENTS WITH LIMIT VALUES THAT REQUIRE MONITORING AT THE WORKPLACE - NOT REQUIRED

EXPOSURE CONTROLS:

APPROPRIATE ENGINEERING CONTROLS - THE USUAL PRECAUTIONARY MEASURES ARE TO BE ADHERED TO WHEN HANDLING CHEMICALS.

PERSONAL PROTECTIVE EQUIPMENT:



HAND PROTECTION	WEAR IMPERMEABLE RUBBER OR PLASTIC GLOVES
EYE PROTECTION	TIGHTLY SEALED SAFETY GOGGLES OR FULL FACE SIDE SHIELDS.
SKIN AND BODY	APRON, COVERALLS AND NON-LEATHER SOLED WORK SHOES. WASH DYE CONTAMINATED CLOTHES AND SKIN WITH MILD SOAP AND DETERGENTS.
RESPIRATORY	WEAR OSHA/NIOSH APPROVED DUST MASK/RESPIRATOR
HYGIENE MEASURES	HANDLE IN ACCORDANCE WITH GOOD INDUSTRIAL HYGIENE AND SAFETY PRACTICES. WASH HANDS AFTER HANDLING MATERIAL.
OTHER PROTECTION	DELUGE SAFETY SHOWER AND EYE WASH STATION SHOULD BE LOCATED NEAR WORK AREA.

SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES

INFORMATION ON BASIC PHYSICAL AND CHEMICAL PROPERTIES :

APPEARANCE, COLOR, ODOR	YELLOW POWDER, NO ODOR
pH	8.0 - 9.0
MELTING POINT/FREEZING POINT	ND
INITIAL BOILING POINT/BOILING RANGE	0.00
FLASHPOINT	NORMALLY STABLE, NOT COMBUSTIBLE NOR FLAMMABLE
EVAPORATION RATE	NO DATA
FLAMMABILITY (SOLID,GAS)	NORMALLY STABLE, NOT COMBUSTIBLE NOR FLAMMABLE
UPPER EXPLOSIVE LIMITS	NA
LOWER EXPLOSIVE LIMITS	NA
VAPOR PRESSURE	NA
VAPOR DENSITY	NA
RELATIVE DENSITY	NA
SOLUBILITY IN WATER	SOLUBLE
PARTITION COEFFICIENT N-OCTANOL/WATER	NO DATA

AUTO-IGNITION TEMPERATURE	NO DATA
DECOMPOSITION TEMPERATURE	NO DATA
VISCOSITY, DYNAMIC	NO DATA
VISCOSITY, CINEMATIC	NO DATA
EXPLOSIVE PROPERTIES	N/A
OXIDIZING PROPERTIES	NA
OTHER INFORMATION	NA

SECTION 10. STABILITY AND REACTIVITY

CHEMICAL STABILITY	STABLE UNDER NORMAL STORAGE AND HANDLING CONDITIONS.
CONDITIONS TO AVOID	OXIDIZING & REDUCING AGENTS MAY DESTROY COLOR.
INCOMPATIBLE MATERIALS	OXIDIZING & REDUCING AGENTS MAY DESTROY COLOR.
HAZARDOUS DECOMPOSITION PRODUCTS	CO, CO2, OXIDES OF NITROGEN AND OTHER POTENTIALLY TOXIC FUMES.

SECTION 11. TOXICOLOGICAL INFORMATION

TOXICOLOGICAL EFFECTS :

ORAL (ANIMAL	GREATER THAN 7,000 MG/KG - RAT
DERMAL (ANIMAL)	NA
EFFECTS TO EYES (ANIMAL)	EYES - RABBIT, NOT IRRITATING
SKIN IRRITATION (ANIMAL)	SKIN - RABBIT, SLIGHT IRRITANT

SKIN CORROSION/IRRITATION	NOT CLASSIFIED
SERIOUS EYE DAMAGE/IRRITATION	CAUSES EYE IRRITATION
RESPIRATORY OR SKIN SENSITIZATION	NOT CLASSIFIED
GERM CELL MUTAGENICITY	NOT CLASSIFIED
CARCINOGENICITY	NOT CLASSIFIED
REPRODUCTIVE TOXICITY	NOT CLASSIFIED
SPECIFIC TARGET ORGAN TOXICITY (SINGLE EXPOSURE)	MAY CAUSE DROWSINESS OR DIZZINESS.
ASPIRATION HAZARD	NOT CLASSIFIED
INHALATION	MAY CAUSE DROWSINESS OR DIZZINESS.
EYE CONTACT	CAUSES SERIOUS EYE IRRITATION.
INGESTION	INGESTION MAY CAUSE NAUSEA, VOMITING AND DIARRHEA

SECTION 12. ECOLOGICAL INFORMATION

TOXICITY	NA
PERSISTENCE AND DEGRADABILITY	NA
BIOACCUMULATIVE POTENTIAL	NA
MOBILITY IN SOIL	LC-50 (LETHAL CONCENTRATION) UG = MICROGRAMS/LITER CHANNEL CATFISH - 2,267,000 UG/LITER RAINBOW TROUT - 1,372,000 UG/LITER BLUEGILL - 3,433,000 UG/LITER
OTHER ADVERSE EFFECTS	NA

SECTION 13. DISPOSAL CONSIDERATION

WASTE DISPOSAL RECOMMENDATION :

EMPTY BAGS THOROUGHLY. CARRY OUT THE PROPER RECYLING, REUSAGE OR DISPOSAL.
PLEASE REFER TO THE RELEVANT EU REGULATIONS, IN PARTICULAR THE GUIDELINES/DECISIONS OF THE COUNCIL REGARDING HANDLING OF WASTES (E.G. 75/442/EEC, 91/689/EEC, 94/67/EC, 94/904/EC) AS IMPLEMENTED IN NATIONAL REGULATIONS.

REGIONAL RECOMMENDATION :

BURY OR INCINERATE ACCORDANCE WITH FEDERAL, STATE AND LOCAL REGULATIONS.

CONTAINERS SHOULD NOT BE REUSED WITHOUT PROFESSIONAL CLEANING AND RECONDITIONING.
OBSERVE ALL LABELED SAFEGUARDS UNTIL CLEANED, RECONDITIONED OR DESTROYED.

PLEASE REFER TO SECTION 8 (EXPOSURE CONTROLS /PERSONAL PROTECTION) OF THIS SDS.

SECTION 14. TRANSPORTATION INFORMATION

UN NUMBER	NONE
UN PROPER SHIPPING NAME	NONE

DEPARTMENT OF TRANSPORTATION (DOT) : NOT HAZARDOUS FOR TRANSPORTATION

TRANSPORT HAZARD CLASS(ES)

HAZARD LABLES (DOT) :

PACKING GROUP (DOT)	NA
DOT SPECIAL PROVISIONS	NA

ADDITIONAL INFORMATION:

OVERLAND TRANSPORT	NONE
TRANSPORT BY SEA	NONE
AIR TRANSPORT	NONE
DOT QUANTITY LIMITATIONS PASSENGER AIRCRAFT	NA
DOT QUANTITY LIMITATIONS CARGO AIRCRAFT	NA

SECTION 15. REGULATORY INFORMATION

US FEDERAL REGULATIONS:

THE SUBSTANCES IS LISTED ON UNITED STATES TSCA (TOXIC SUBSTANCE CONTROL ACT) INVENTORY.

US STATE REGULATIONS:

NONE

CHEMICAL IDENTITY:

518-47-8	TSCA	DSL	NDSL	EINECS	ELINCS	ENCS	CHINA	KECL	PICCS	AICS
	X	-	-	-	-	-	-	-	-	-

TSCA STATUS	IN COMPLIANCE
E C CLASSIFICATION	(67/548/EEC - 88/379/EEC) N/A
EINECS NUMBER	
REACH CLASSIFICATION	
R PHRASES	
ADDITIONAL REGULATORY INFORMATION	

SECTION 16. OTHER INFORMATION

INDICATION OF CHANGES:

NA

OTHER INFORMATION:

NA

GHS FULL TEXT PHRASES:

MAY BE HARMFUL IF INHALED	H333
CAUSES EYE IRRITATION	H320
CASUES MILD SKIN IRRITATION	H316

	HEALTH	FLAMMABILITY	REACTIVITY	PERSONAL PROT
H. M. I. S. CLASSIFICATION:	1	0	0	D

HMIS CODE: **4** - SEVERE HAZARD, **3** - SERIOUS HAZARD, **2** - MODERATE HAZARD, **1** - SLIGHT HAZARD, **0** - MINIMAL HAZARD

SAFETY DATA SHEET (SDS)
REVISION DATE: 03/03/2016

ALL INFORMATION AND DATA APPEARING ON THIS SDS ARE BELIEVED TO BE RELIABLE AND ACCURATE. HOWEVER, IT IS THE USER' S RESPONSIBILITY TO DETERMINE THE SAFETY, TOXICITY, AND SUITABILITY FOR USE OF THE PRODUCT DESCRIBED. SINCE THE ACTUAL USE BY OTHERS IS BEYOND OUR CONTROL, NO GUARANTEE, EXPRESSED OR IMPLIED, IS MADE BY HUE CORPORATION.
USER ASSUMES ALL RISK AND RESPONSIBILITY.

ATTACHMENT 3

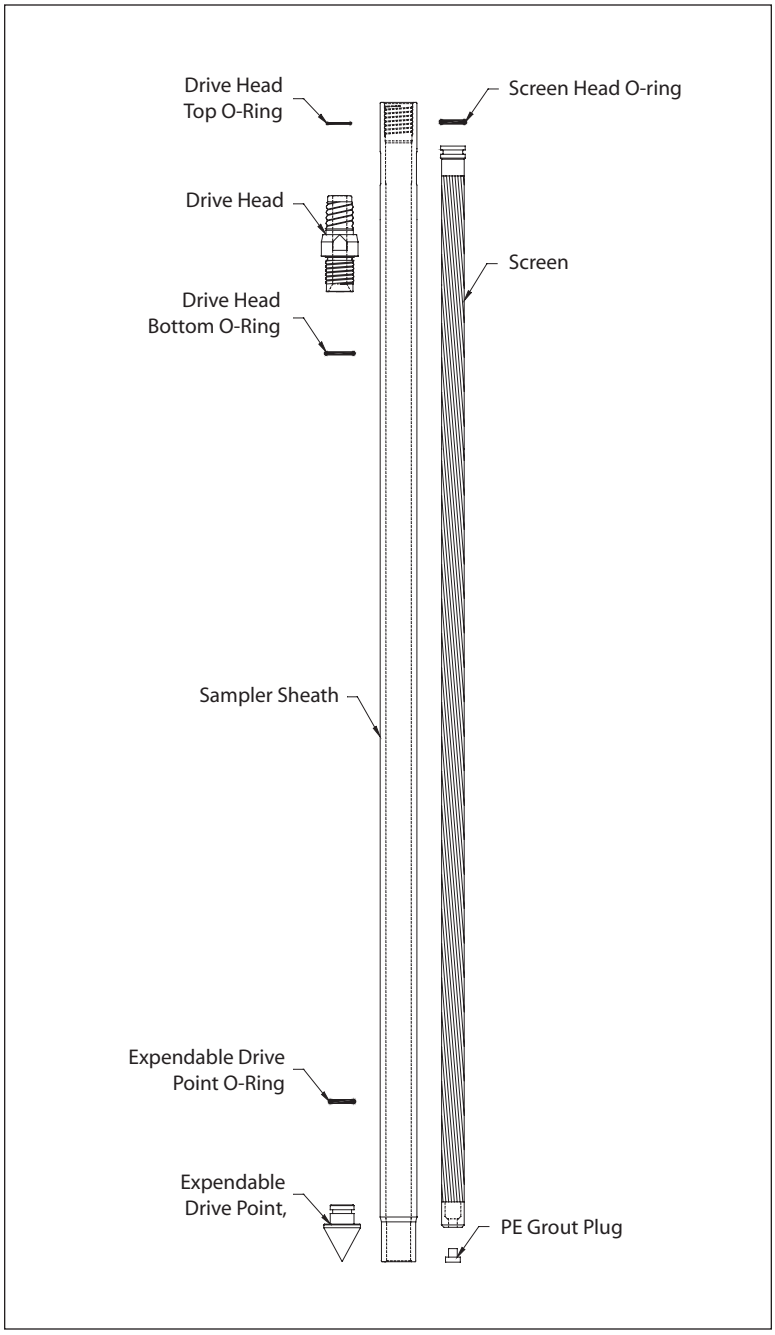
SOPs Geoprobe® Screen Point 16 and Screen Point 22 Groundwater Samplers

GEOPROBE® SCREEN POINT 16 GROUNDWATER SAMPLER

STANDARD OPERATING PROCEDURE

Technical Bulletin No. MK3142

PREPARED: November, 2006



GEOPROBE® SCREEN POINT 16 GROUNDWATER SAMPLER PARTS



Geoprobe® and Geoprobe Systems®, Macro-Core® and Direct Image® are Registered Trademarks of Kejr, Inc., Salina, Kansas

Screen Point 16 Groundwater Sampler is manufactured under U.S. Patent 5,612,498

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1.0 OBJECTIVE

The objective of this procedure is to drive a sealed stainless steel or PVC screen to depth, deploy the screen, obtain a representative water sample from the screen interval, and grout the probe hole during abandonment. The Screen Point 16 Groundwater Sampler enables the operator to conduct abandonment grouting that meets American Society for Testing and Materials (ASTM) Method D 5299 requirements for decommissioning wells and borings for environmental activities (ASTM 1993).

2.0 BACKGROUND

2.1 Definitions

Geoprobe®: A brand name of high quality, hydraulically powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and monitoring, soil conductivity and contaminant logging, grouting, and materials injection.

Screen Point 16 (SP16) Groundwater Sampler: A direct push device consisting of a PVC or stainless steel screen that is driven to depth within a sealed, steel sheath and then deployed for the collection of representative groundwater samples. The assembled SP16 Sampler is approximately 51.5 inches (1308 mm) long with an OD of 1.625 inches (41 mm). Upon deployment, up to 41 inches (1041 mm) of screen can be exposed to the formation. The Screen Point 16 Groundwater Sampler is designed for use with 1.5-inch probe rods and machines equipped with the more powerful GH60 Hydraulic Hammer. Operators with GH40 Series hammers may chose to use this sampler in soils where driving is difficult.

Rod Grip Pull System: An attachment mounted on the hydraulic hammer of a direct push machine which makes it possible to retract the tool string with extension rods or flexible tubing protruding from the top of the probe rods. The Rod Grip Pull System includes a pull block with rod grip jaws that are bolted directly to the machine. A removable handle assembly straddles the tool string while hooking onto the pull block to effectively grip the probe rods as the hammer is raised. A separate handle assembly is required for each probe rod diameter.

2.2 Discussion

In this procedure, the assembled Screen Point 16 Groundwater Sampler (Fig. 2.1A) is threaded onto the leading end of a Geoprobe® probe rod and advanced into the subsurface with a Geoprobe® direct push machine. Additional probe rods are added incrementally and advanced until the desired sampling interval is reached. While the sampler is advanced to depth, O-ring seals at each rod joint, the drive head, and the expendable drive point provide a watertight system. This system eliminates the threat of formation fluids entering the screen before deployment and assures sample integrity.

Once at the desired sampling interval, extension rods are sent downhole until the leading rod contacts the bottom of the sampler screen. The tool string is then retracted approximately 44 inches (1118 mm) while the screen is held in place with the extension rods (Fig. 2.1B). As the tool string is retracted, the expendable point is released from the sampler sheath. The tool string and sheath may be retracted the full length of the screen or as little as a few inches if a small sampling interval is desired.

There are three types of screens that can be used in the Screen Point 16 Groundwater Sampler. Two of the these, a stainless steel screen with a standard slot size of 0.004 inches (0.10 mm) and a PVC screen with a standard slot size of 0.010 inches (0.25 mm), are recovered with the tool string after sampling. The third screen is also manufactured from PVC with a standard slot size of 0.010 inches (0.25 mm), but is designed to be left downhole when sampling is complete. This disposable screen has an exposed screen length of approximately 43 inches (1092 mm). The two screens that are recovered with the sampler both have an exposed screen length of approximately 41 inches (1041 mm).

(continued on following page)

An O-ring on the head of the stainless steel screens maintains a seal at the top of the screen. As a result, any liquid entering the sampler during screen deployment must first pass through the screen. PVC screens do not require an O-ring because the tolerance between the screen head and sampler sheath is near that of the screen slot size.

The screens are constructed such that flexible tubing, a mini-bailer, or a small-diameter bladder pump can be inserted into the screen cavity. This makes direct sampling possible from anywhere within the saturated zone. A removable plug in the lower end of the screens allows the user to grout as the sampler is extracted for further use.

Groundwater samples can be obtained in a number of ways. A common method utilizes polyethylene (TB25L) or Teflon® (TB25T) tubing and a Check Valve Assembly (GW4210). The check valve (with check ball) is attached to one end of the tubing and inserted down the casing until it is immersed in groundwater. Water is pumped through the tubing and to the ground surface by oscillating the tubing up and down.

An alternative means of collecting groundwater samples is to attach a peristaltic or vacuum pump to the tubing. This method is limited in that water can be pumped to the surface from a maximum depth of approximately 26 feet (8 m). Another technique for groundwater sampling is to use a stainless steel Mini-Bailer Assembly (GW41). The mini-bailer is lowered down the inside of the casing below the water level where it fills with water and is then retrieved from the casing.

The latest option for collecting groundwater from the SP16 sampler is to utilize a Geoprobe® MB470 Series Mechanical Bladder Pump (MBP)*. The MBP may be used to meet requirements of the low-flow sampling protocol (Puls and Barcelona 1996, ASTM 2003). Through participation in a U.S. EPA Environmental Technology Verification study, it was confirmed that the MB470 can provide representative samples (EPA 2003).

**The Mechanical Bladder Pump is manufactured under U.S. Patent No. 6,877,965 issued April 12, 2005.*

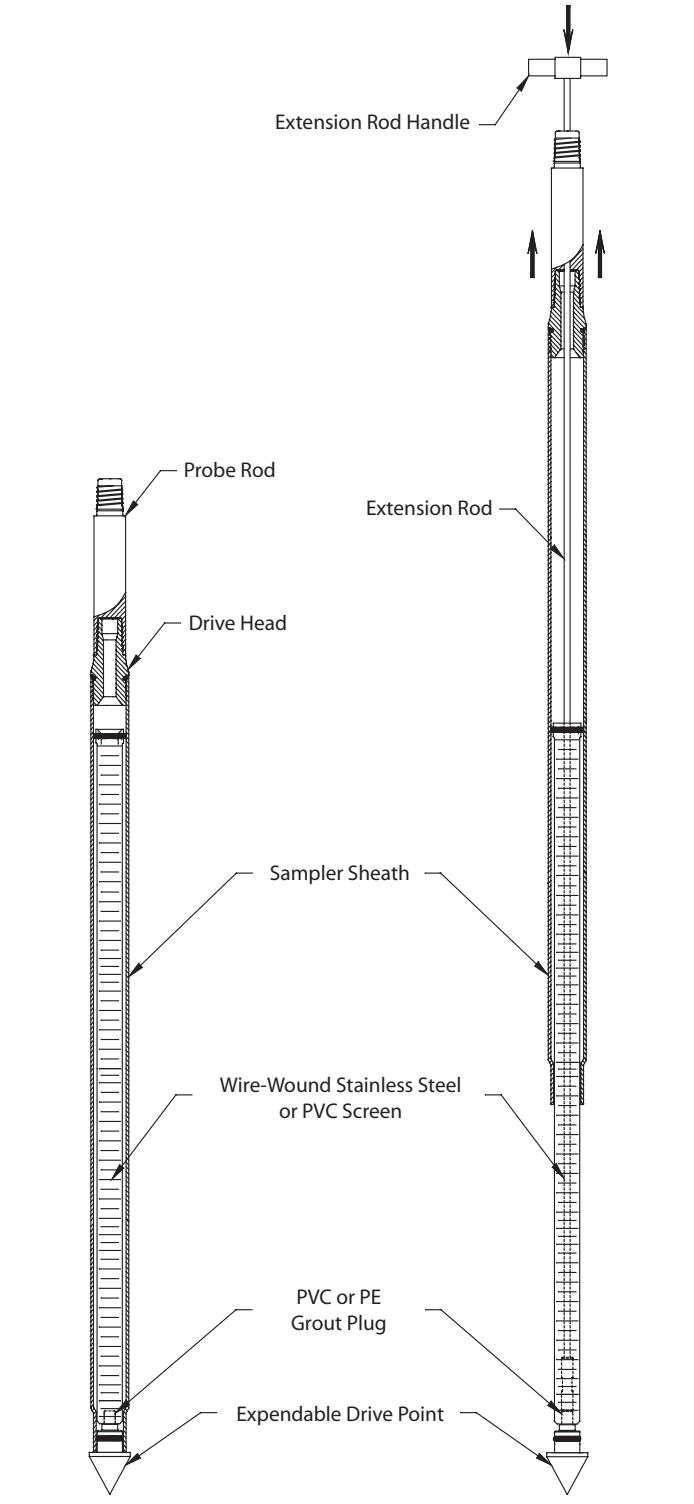


FIGURE 2.1
Screen Point 16 Groundwater Sampler

3.0 TOOLS AND EQUIPMENT

The following tools and equipment can be used to successfully recover representative groundwater samples with the Geoprobe® Screen Point 16 Groundwater Sampler. Refer to Figures 3.1 and 3.2 for identification of the specified parts. Tools are listed below for the most common SP16 / 1.5-inch probe rod configurations. Additional parts for optional rod sizes and accessories are listed in Appendix A.

SP16 Sampler Parts	**Part Number**
SP16 Sampler Sheath	15187
SP16 Drive Head, 0.5-inch bore, 1.5-inch rods*	18307
SP16 O-ring Service Kit, 1.5-inch rods *(includes 4 each of the O-ring packets below)*	15844
O-rings for Top of SP16 Drive Head, 1.5-inch rods only (Pkt. of 25)	*15389*
O-rings for Bottom of SP16 Drive Head (Pkt. of 25)	*13196*
O-rings for GW1520 Screen Head (Pkt. of 25)	*GW1520R*
O-rings for SP16 Expendable Drive Point (Pkt. of 25)	*GW1555R*
Screen, Wire-Wound Stainless Steel, 4-Slot*	GW1520
Grout Plugs, PE (Pkg. of 25)	GW1552K
Expendable Drive Points, steel, 1.625-inch OD (Pkg. of 25)*	GW1555K
Screen Point 16 Groundwater Sampler Kit, 1.5-inch Probe Rods *(includes 1 each of: 15187, 18307, 15844, GW1520, GW1535, GW1540, GW1555K, and GW1552K)*	15770

Probe Rods and Probe Rod Accessories	**Part Number**
Drive Cap, 1.5-inch probe rods, threadless, (for GH60 Hammer)	12787
Pull Cap, 1.5-inch probe rods	15090
Probe Rod, 1.5-inch x 60-inch*	11121

Extension Rods and Extension Rod Accessories	**Part Number**
Screen Push Adapter	GW1535
Grout Plug Push Adapter	GW1540
Extension Rod, 60-inch*	10073
Extension Rod Coupler	AT68
Extension Rod Handle	AT69
Extension Rod Jig	AT690
Extension Rod Quick Link Coupler, pin	AT695
Extension Rod Quick Link Coupler, box	AT696

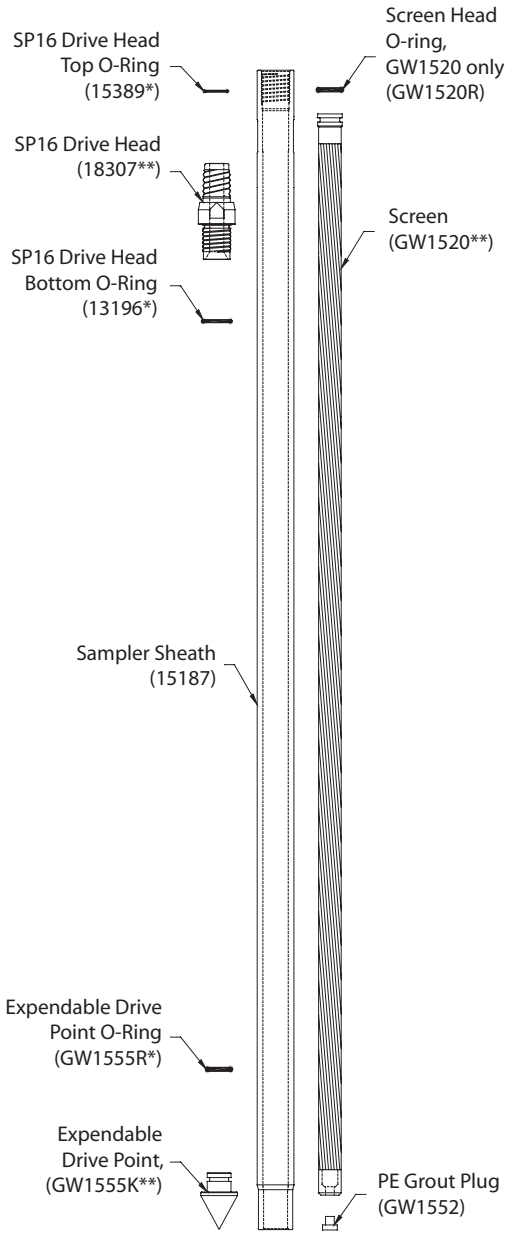
Grout Accessories	**Part Number**
Grout Nozzle, for 0.375-inch OD tubing	GW1545
High-Pressure Nylon Tubing, 0.375-inch OD / 0.25-inch ID, 100-ft. (30 m)	11633
Grout Machine, self-contained*	GS1000
Grout System Accessories Package, 1.5-inch rods	GS1015

Groundwater Purging and Sampling Accessories	**Part Number**
Polyethylene Tubing, 0.375-inch OD, 500 ft.*	TB25L
Check Valve Assembly, 0.375-inch OD Tubing*	GW4210
Water Level Meter, 0.438-inch OD Probe, 100 ft. cable*	GW2000
Mechanical Bladder Pump**	MB470
Mini Bailer Assembly, stainless steel	GW41

Additional Tools	**Part Number**
Adjustable Wrench, 6.0-inch	FA200
Adjustable Wrench, 10.0-inch	FA201
Pipe Wrenches	NA

* See Appendix A for additional tooling options.

** Refer to the Standard Operating Procedure (SOP) for the Mechanical Bladder Pump (Technical Bulletin No. MK3013) for additional tooling needs.



* Included in the SP16 O-ring Service Kit (15844)
** Part numbers show most common tool configuration. See Appendix A for alternative parts.

FIGURE 3.1
SP16 Sampler Parts

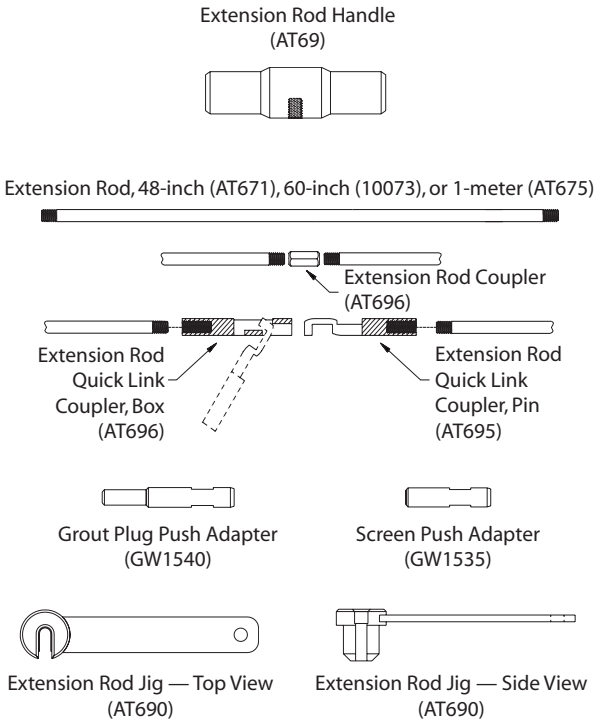


FIGURE 3.2
Geoprobe® Extension Rods and Accessories

4.0 OPERATION

4.1 Basic Operation

The SP16 sampler utilize a stainless steel or PVC screen which is encased in an alloy steel sampler sheath. An expendable drive point is placed in the lower end of the sheath while a drive head is attached to the top. O-rings on the drive head and expendable point provide a watertight sheath which keeps contaminants out of the system as the sampler is driven to depth.

Once the sampling interval is reached, extension rods equipped with a screen push adapter are inserted down the ID of the probe rods. The tool string is then retracted up to 44 inches (1118 mm) while the screen is held in place with the extension rods. The system is now ready for groundwater sampling. When sampling is complete, a removable plug in the bottom of the screen allows for grouting below the sampler as the tool string is retrieved.

4.2 Sampler Options

The Screen Point 15 and Screen Point 16 Groundwater Samplers are nearly identical. Subtle differences in the design of the SP16 sampler make it more durable than the earlier SP15 system. Operators of GH60-equipped machines should always utilize SP16 tooling. Operators of machines equipped with GH40 Series hammers may also choose SP16 tooling when sampling in difficult probing conditions.

A 1.75-inch OD Expendable Drive Point (17066K) and Disposable PVC Screen (16089) provide two useful options for the SP16 sampler. The 1.75-inch drive point may be used when soil conditions make it difficult to remove the sampler after driving to depth. The disposable PVC screen may be left downhole after sampling (when regulations permit) to eliminate the time required for screen decontamination.

4.3 Decontamination

In order to collect representative groundwater samples, all sampler parts must be thoroughly cleaned before and after each use. Scrub all metal parts using a stiff brush and a nonphosphate soap solution. Steam cleaning may be substituted for hand-washing if available. Rinse with distilled water and allow to air-dry before assembly.

4.4 SP16 Sampler Assembly (Figure 4.1)

Part numbers are listed for a standard SP16 sampler using 1.5-inch probe rods. Refer to Page 6 for screen and drive head alternatives.

1. Place an O-ring on a steel expendable drive point (GW1555K). Firmly seat the expendable point in the necked end of a sampler sheath (15187).

2. Install a PE Grout Plug (GW1552) in the bottom end of a Wire-wound Stainless Steel Screen (GW1520). Place a GW1520R O-ring in the groove on the top end of the screen.

3. Slide the screen inside of the sampler sheath with the grout plug toward the bottom of the sampler. Ensure that the expendable point was not displaced by the screen.

4. Install a bottom O-ring (13196) on a Drive Head (18307 or 15188). Thread the drive head into the sampler sheath using an adjustable wrench if necessary to ensure complete engagement of the threads. Attach a Drive Cap (12787 or 15590) to the top of the drive head.

   NOTE: The 18307 drive head should be used whenever possible as the smaller 0.5-inch ID provides a greater material cross-section for increased durability.

Sampler assembly is complete.

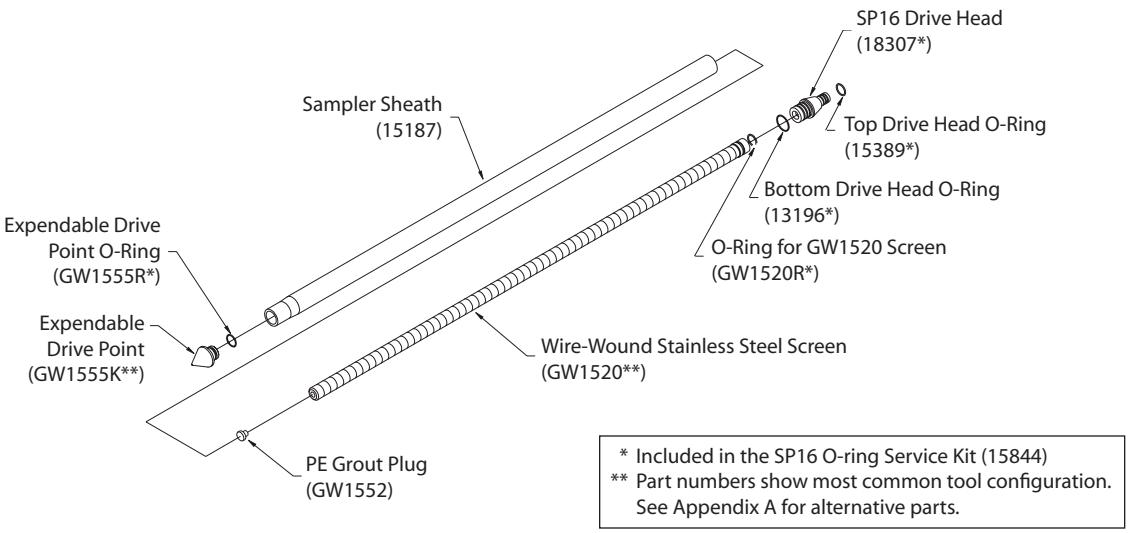


FIGURE 4.1
Screen Point 16 Groundwater Sampler Assembly

4.5 Advancing the SP16 Sampler

To provide adequate room for screen deployment with the Rod Grip Pull System, the probe derrick should be extended a little over halfway out of the carrier vehicle when positioning for operation.

1. Begin by placing the assembled sampler (Fig. 2.1.A) in the driving position beneath the hydraulic hammer of the direct push machine as shown in Figure 4.2.

2. Advance the sampler with the throttle control at slow speed for the first few feet to ensure that the sampler is aligned properly. Switch to fast speed for the remainder of the probe stroke.

3. Completely raise the hammer assembly. Remove the drive cap and place an O-ring in the top groove of the drive head. Distilled water may be used to lubricate the O-ring if needed.

Add a probe rod (length to be determined by operator) and reattach the drive cap to the rod string. Drive the sampler the entire length of the new rod with the throttle control at fast speed.

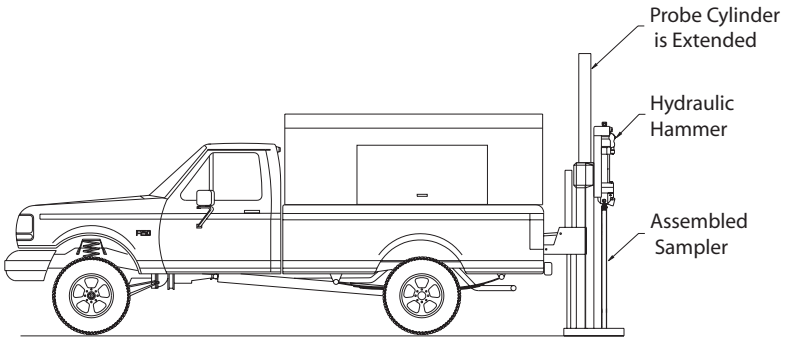


FIGURE 4.2
Screen Point 16 Groundwater Sampler in Driving Position

4. Repeat Step 3 until the desired sampling interval is reached. Approximately 12 inches (305 mm) of the last probe rod must extend above the ground surface to allow attachment of the puller assembly. A 12-inch (305 mm) rod may be added if the tool string is over-driven.

5. Remove the drive cap and retract the probe derrick away from the tool string.

4.6 Screen Deployment

1. Thread a screen push adapter (GW1535) on an extension rod of suitable length (AT671, 10073, or AT675). Attach a threaded coupler (AT68) to the other end of the extension rod. Lower the extension rod inside of the probe rod taking care not to drop it down the tool string. An extension rod jig (AT690) may be used to hold the rods.

2. Add extension rods until the adapter contacts the bottom of the screen. To speed up this step, it is recommended that Extension Rod Quick Links (AT695 and AT696) are used at every other rod joint.

3. Ensure that at least 48 inches (1219 mm) of extension rod protrudes from the probe rod. Thread an extension rod handle (AT69) on the top extension rod.

4. Maneuver the probe assembly into position for pulling.

5. Raise (pull) the tool string while physically holding the screen in place with the extension rods (Fig. 4.3.B). A slight knock with the extension rod string will help to dislodge the expendable point and start the screen moving inside the sheath.

   Raise the hammer and tool string about 44 inches (1118 cm) if using a GW1520 or GW1530 screen. At this point the screen head will contact the necked portion of the sampler sheath (Fig. 4.3.C.) and the extension rods will rise with the probe rods. Use care when deploying a PVC screen so as not to break the screen when it contacts the bottom of the sampler sheath.

   The Disposable Screen (16089) will extend completely out of the sheath if the tool string is raised more than 45 inches (1143 mm). Measure and mark this distance on the top extension rod to avoid losing the screen during deployment.

6. Remove the rod grip handle, lower the hammer assembly, and retract the probe derrick. Remove the top extension rod (with handle) and top probe rod. Finally, extract all extension rods.

7. Groundwater samples can now be collected with a mini-bailer, peristaltic or vacuum pump, tubing bottom check valve assembly, bladder pump, or other acceptable small diameter sampling device.

   When inserting tubing or a bladder pump down the rod string, ensure that it enters the screen interval. The leading end of the tubing or bladder pump will sometimes catch at the screen head giving the illusion that the bottom of the screen has been reached. An up-and-down motion combined with rotation helps move the tubing or bladder pump past the lip and into the screen.

4.7 Abandonment Grouting for GW1520 and GW1530 Screens

The SP16 Sampler can meet ASTM D 5299 requirements for abandoning environmental wells or borings when grouting is conducted properly. A removable grout plug makes it possible to deploy tubing through the bottom of GW1520 and GW1530 screens. A GS500 or GS1000 Grout Machine is then used to pump grout into the open probe hole as the sampler is withdrawn. The following procedure is presented as an example only and should be modified to satisfy local abandonment grouting regulations.

1. Maneuver the probe assembly into position for pulling. Attach the rod grip puller to the top probe rod. Raise the tool string approximately 4 to 6 inches (102 to 152 cm) to allow removal of the grout plug.

2. Thread the Grout Plug Push Adapter (GW1540) onto an extension rod. Insert the adapter and extension rod inside the probe rod string. Add extension rods until the adapter contacts the grout plug at the bottom of the screen. Attach the handle to the top extension rod. When the extension rods are slightly raised and lowered, a relatively soft rebound should be felt as the adapter contacts the grout plug. This is especially true when using a PVC screen.

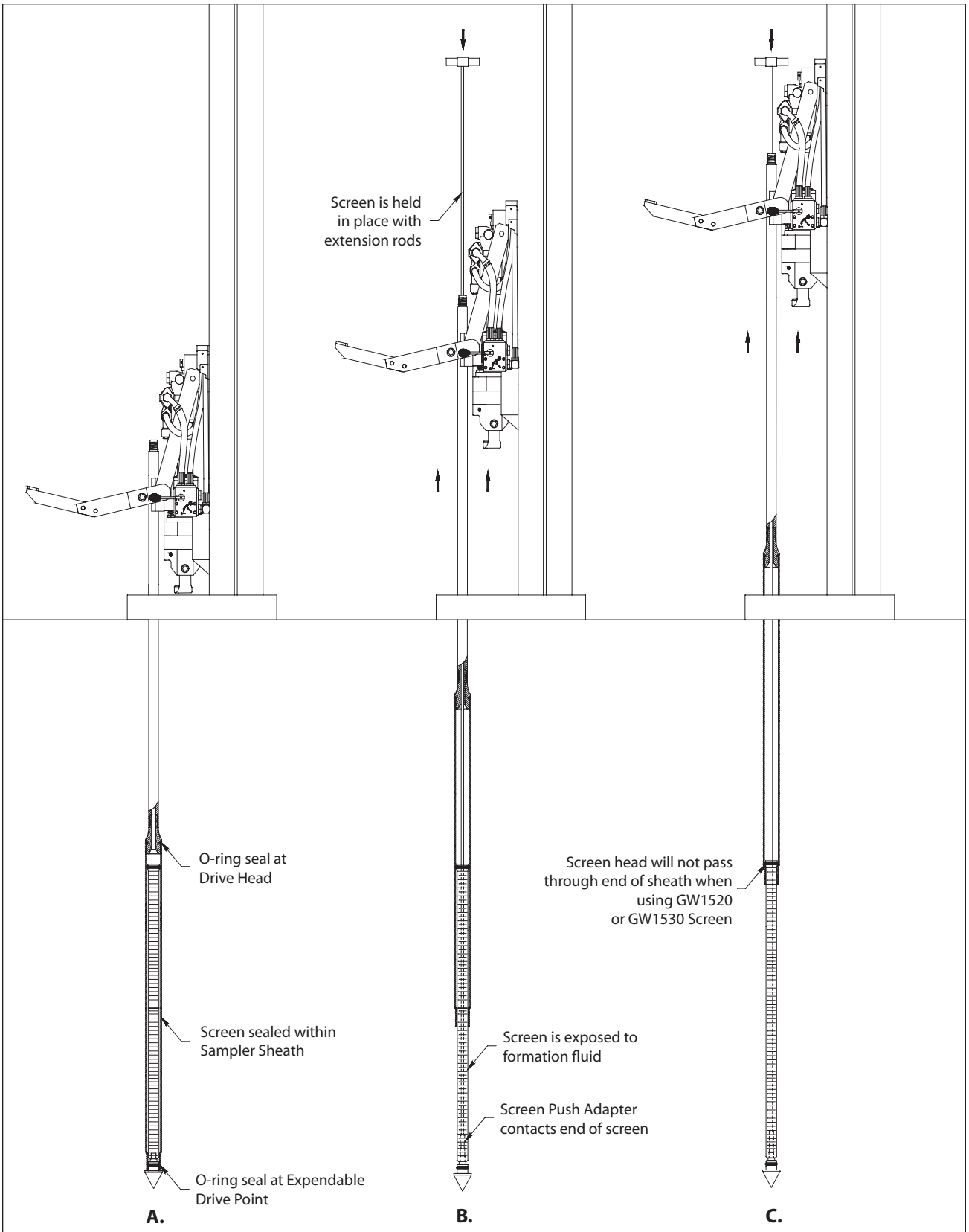


FIGURE 4.3
Screen Deployment for SP16 Sampler

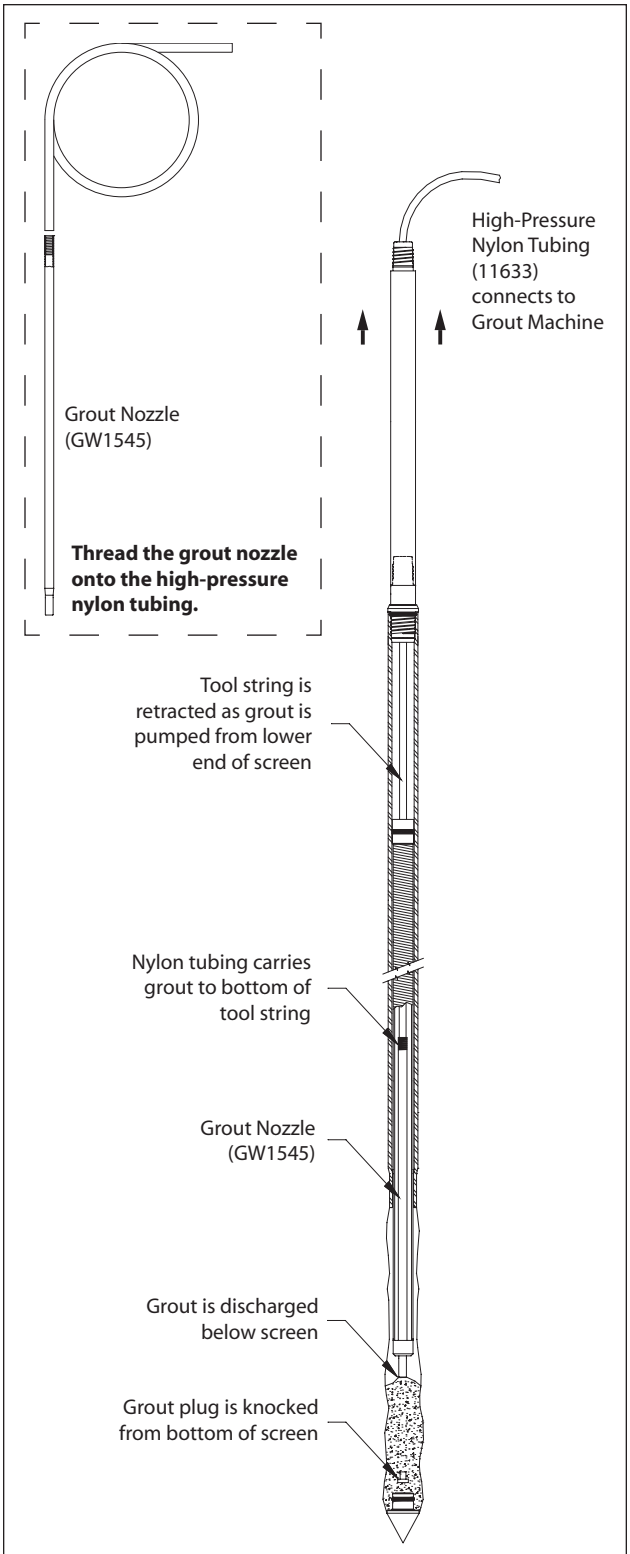


FIGURE 4.4
Grouting the SP16 Sampler With a GW1520 or GW1530 Screen

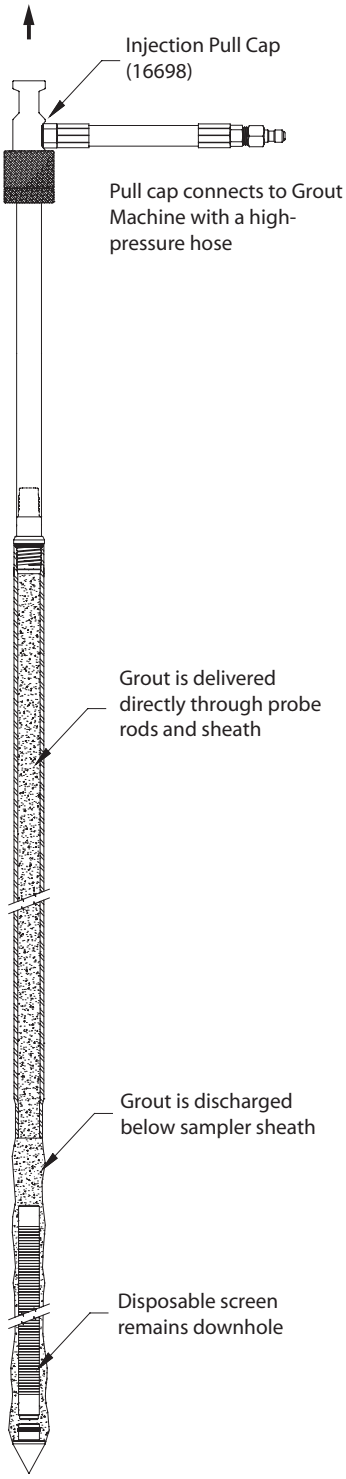


FIGURE 4.5
Grouting the SP16 Sampler With a 16089 Disposable Screen

3. Place a mark on the extension rod even with the top of the probe rod. Apply downward pressure on the extension rods and push the grout plug out of the screen. The mark placed on the extension rod should now be below the top of the probe rod. Remove all extension rods.

   Note: When working with a stainless steel screen, it may be necessary to raise and quickly lower the extension rods to jar the grout plug free. When the plug is successfully removed, a metal-on-metal sensation may be noted as the extension rods are gently "bounced" within the probe rods.

4. A Grout Nozzle (GW1545) is now connected to High-Pressure Nylon Tubing (11633) and inserted down through the probe rods to the bottom of the screen (Fig. 4.4). It may be necessary to pump a small amount of clean water through the tubing during deployment to jet out sediments that settled in the bottom of the screen. Resistance will sometimes be felt as the grout nozzle passes through the drive head. Rotate the tubing while moving it up-and-down to ensure that the nozzle has reached the bottom of the screen and is not hung up on the drive head.

   Note: *All probe rods remain strung on the tubing as the tool string is pulled. Provide extra tubing length to allow sufficient room to lay the rods on the ground as they are removed. An additional 20 feet is generally enough.*

5. Operate the grout pump while pulling the first rod with the rod grip pull system. Coordinate pumping and pulling rates so that grout fills the void left by the sampler. After pulling the first rod, release the rod grip handle, fully lower the hammer, and regrip the tool string. Unthread the top probe and slide it over the tubing placing it on the ground near the end of the tubing.

6. Repeat Step 5 until the sampler is retrieved. Do not bend or kink the tubing when pulling and laying out the probe rods. Sharp bends create weak spots in the tubing which may burst when pumping grout. Remember to operate the grout pump only when pulling the rod string. The probe hole is thus filled with grout from the bottom up as the rods are extracted.

7. Promptly clean all probe rods and sampler parts before the grout sets up and clogs the equipment.

4.8 Abandonment Grouting for the 16089 Disposable Screen

ASTM D 5299 requirements can also be met for the SP16 samplers when using the 16089 disposable screen. Because the screen remains downhole after sampling, the operator may choose either to deliver grout to the bottom of the tool string with nylon tubing or pump grout directly through the probe rods using an Injection Pull Cap (16698). A GS500 or GS1000 Grout Machine is needed to pump grout into the open probe hole as the sampler is withdrawn. The following procedure is presented as an example only and should be modified to satisfy local abandonment grouting regulations.

1. Maneuver the probe assembly into position for pulling with the rod grip puller.

2. Thread the screen push adapter onto an extension rod. Insert the adapter and extension rod inside the probe rod string. Add extension rods until the adapter contacts the bottom of the screen. Attach the handle to the top extension rod.

3. The disposable screen must be extended at least 46 inches (1168 mm) to clear the bottom of the sampler sheath. Considering the length of screen deployed in Section 4.7, determine the remaining distance required to fully extend the screen from the sheath. Mark this distance on the top extension rod.

4. Pull the tool string up to the mark on the top extension rod while holding the disposable screen in place.

   The screen is now fully deployed and the sampler is ready for abandonment grouting. Apply grout to the bottom of the tool string during retrieval using either flexible tubing (as described in Section 4.7) or an injection pull cap (Fig. 4.5). This section continues with a description of grouting with a pull cap.

5. Remove the rod grip handle and maneuver the probe assembly directly over the tool string. Thread an Injection Pull Cap (16698) onto the top probe rod and close the hammer pull latch over the top of the pull cap.

6. Connect the pull cap to a Geoprobe® grout machine using a high-pressure grout hose.

7. Operate the pump to fill the entire tool string with grout. When a sufficient volume has been pumped to fill the tool string, begin pulling the rods and sampler while continuing to operate the grout pump. Considering the known pump volume and sampler cross-section, time tooling withdrawal to slightly "overpump" grout into the subsurface. This will ensure that all voids are filled during sampler retrieval.

   The grouting process can lubricate the probe hole sufficiently to cause the tool string to slide back downhole when disconnected from the pull cap. Prevent this by withdrawing the tool string with the rod grip puller while maintaining a connection to the grout machine with the pull cap.

4.9 Retrieving the Screen Point 16 Sampler

If grouting is not required, the Screen Point 16 Sampler can be retrieved by pulling the probe rods as with most other Geoprobe® applications. The Rod Grip Pull System should be used for this process as it allows the operator to remove rods without completely releasing the tool string. This avoids having the probe rods fall back downhole when released during the pulling procedure. A standard Pull Cap (15164) may still be used if preferred. Refer to the Owner's Manual for your Geoprobe® direct push machine for specific instructions on pulling the tool string.

5.0 REFERENCES

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Puls, Robert W., and Michael J. Barcelona, 1996. Ground Water Issue: Low-Flow (Minimal Drawdown) Ground Water Sampling Procedures. EPA/540/S-95/504. April.

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Appendix A
ALTERNATIVE PARTS

The following parts are available to meet unique soil conditions. See section 3.0 for a complete listing of the common tool configurations for the Geoprobe® Screen Point 16 Groundwater Sampler.

SP16 Sampler Parts and Accessories	**Part Number**
SP16 Drive Head, 0.625-inch bore, 1.5-inch rods	15188
Expendable Drive Points, aluminum, 1.625-inch OD (Pkg. of 25)	GW1555ALK
Expendable Drive Points, steel, 1.75-inch OD (Pkg. of 25)	17066K
Screen, PVC, 10-Slot	GW1530
Screen, Disposable, PVC, 10-Slot	16089

Groundwater Purging and Sampling Accessories	**Part Number**
Polyethylene Tubing, 0.25-inch OD, 500 ft	TB17L
Polyethylene Tubing, 0.5-inch OD, 500 ft	TB37L
Polyethylene Tubing, 0.625-inch OD, 50 ft	TB50L
Check Valve Assembly, 0.25-inch OD Tubing	GW4240
Check Valve Assembly, 0.5-inch OD Tubing	GW4220
Check Valve Assembly, 0.625-inch OD Tubing	GW4230
Water Level Meter, 0.375-inch OD Probe, 100-ft. cable	GW2001
Water Level Meter, 0.438-inch OD Probe, 200-ft. cable	GW2002
Water Level Meter, 0.375-inch OD Probe, 200-ft. cable	GW2003
Water Level Meter, 0.438-inch OD Probe, 30-m cable	GW2005
Water Level Meter, 0.438-inch OD Probe, 60-m cable	GW2007
Water Level Meter, 0.375-inch OD Probe, 60-m cable	GE2008

Grouting Accessories	**Part Number**
Grout Machine, auxiliary-powered	GS500

Probe Rods, Extension Rods, and Accessories	**Part Number**
Probe Rod, 1.5-inch x 1-meter	17899
Probe Rod, 1.5-inch x 48-inch	13359
Drive Cap, 1.5-inch rods (for GH40 Series Hammer)	15590
Rod Grip Pull Handle, 1.5-inch Probe Rods (for GH40 Series Hammer)	GH1555
Extension Rod, 48-inch	AT671
Extension Rod, 1-meter	AT675

Equipment and tool specifications, including weights, dimensions, materials, and operating specifications included in this brochure are subject to change without notice. Where specifications are critical to your application, please consult Geoprobe Systems®.



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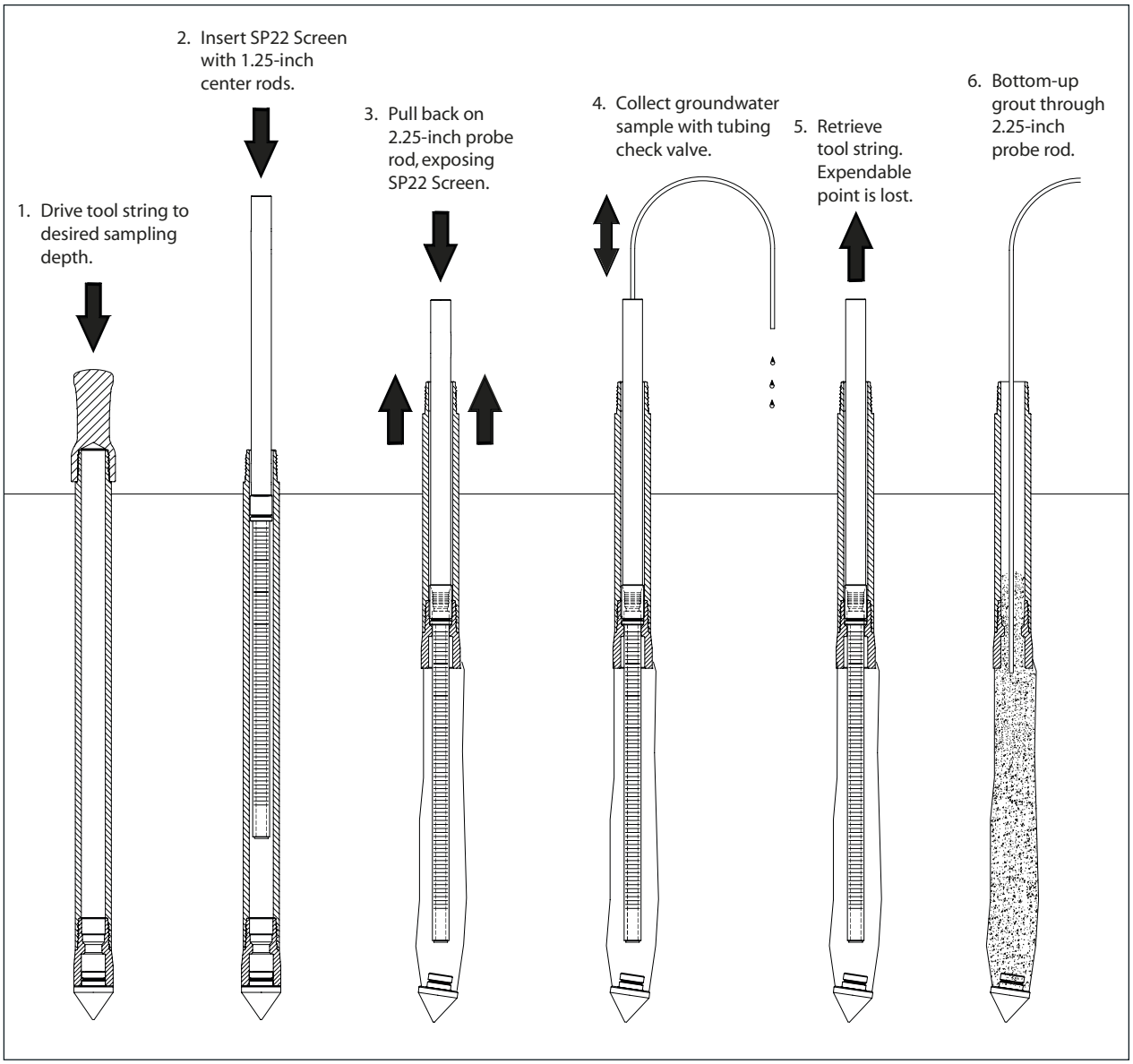
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GEOPROBE® SCREEN POINT 22 GROUNDWATER SAMPLER

STANDARD OPERATING PROCEDURE

Technical Bulletin No. MK3173

PREPARED: April 2010



OPERATION OF THE GEOPROBE® SCREEN POINT 22 GROUNDWATER SAMPLER



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Screen Point 22 Groundwater Sampler is manufactured under U.S. Patent 5,612,498

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1.0 OBJECTIVE

The objective of this procedure is to deploy a stainless steel or PVC screen at depth, obtain a representative water sample from the screen interval, and grout the probe hole during abandonment. The Screen Point 22 Groundwater Sampler enables the operator to conduct abandonment grouting that meets American Society for Testing and Materials (ASTM) Method D 5299 requirements for decommissioning wells and borings for environmental activities (ASTM 1993).

2.0 BACKGROUND

2.1 Definitions

Geoprobe®: A brand name of high quality, hydraulically powered machines that utilize static force and percussion or rotation to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform activities such as soil core and soil gas sampling, groundwater sampling and monitoring, soil conductivity and contaminant logging, grouting, and materials injection.

Screen Point 22 (SP22) Groundwater Sampler: A direct push device consisting of a PVC or stainless steel screen that is lowered (post-run) to depth within a sealed string of steel probe rods and then deployed for the collection of representative groundwater samples. Upon deployment, up to 48 inches (1219 mm) of screen can be exposed to the formation. There is also an optional 12-inch screen that can be used. The Screen Point 22 Groundwater Sampler is designed for use with 2.25-inch probe rods and machines equipped with the more powerful GH60 and GH80 series hydraulic hammers. Operators with GH40 series hammers may choose to use this sampler in soils where driving is easier.

Rod Grip Pull System: An attachment mounted on the hydraulic hammer of a direct push machine which makes it possible to retract the tool string with probe rods or fiexible tubing protruding from the top of the probe rods. The Rod Grip Pull System includes a pull block with rod grip jaws that are bolted directly to the machine. A removable handle assembly straddles the tool string while hooking onto the pull block to effectively grip the probe rods as the hammer is raised. A separate handle assembly is required for each probe rod diameter.

2.2 Discussion (Fig. 2.1)

In this procedure, 2.25-inch probe rods are advanced into the subsurface with a Geoprobe® subsurface machine (Fig. 2.1, Step 1). While the tool string is advanced to depth, O-ring seals at each rod joint, the expendable point holder, and the expendable drive point provide a watertight system. This eliminates the threat of formation fiuids entering the screen before deployment and assures sample integrity.

Once the leading end of the 2.25-inch probe rods reaches the desired sampling interval, an SP22 screen is lowered to the bottom of the rods using a string of either 1.25-inch outside diameter (OD) light-weight center rods, 1.25-inch probe rods, or 0.75-inch schedule 40 fiush-thread PVC riser (Fig. 2.1, Step 2). The 2.25-inch rods are then retracted while the SP22 screen is held in place with the 1.25-inch rods or PVC riser (Fig 2.1, Step 3). As the 2.25-inch tool string is retracted, the expendable point is released from the expendable point holder. The tool string and expendable point holder may be retracted the full length of the screen or as little as a few inches if a small sampling interval is desired.

The SP22 Sampler can also be used with the Geoprobe® DT22 system. (Fig. 2.2)

(continued on following page)

Expendable Drive Points
The SP22 system utilizes an SP22 Expendable Point Holder (33764) and standard 2.45-inch (62-mm) OD steel Expendable Drive Points for 2.25-inch probe rods (AT2015K). Extended Shank Expendable Drive Points (19442) are available for soft soil conditions where standard points may be advanced out of the point holder during percussion. A third option is to use a part number 43128 SP22 Expendable Point Holder along with 1.625-inch (41-mm) steel Expendable Drive Points (GW1555K). These smaller drive points are more economical to purchase and ship, but must not be used with GH80 Series Hydraulic Hammers as they may not stay seated during percussion.

Screens
Two types of screens have been developed for use in the Screen Point 22 Groundwater Sampler - a stainless steel screen with a standard slot size of 0.004 inches (0.10 mm) and a PVC screen with a standard slot size of 0.010 inches (0.25 mm). These screens are available in nominal 48- and 12-inch lengths. Effective screen lengths for the 48- and 12-inch PVC screens are 48 inches (1219 mm) and 12 inches (305 mm), while 48- and 12-inch stainless steel screens have effective screen lengths of 43 inches (1092 mm) and 14 inches (356 mm) respectively. Both types of screens are recovered with the tool string after sampling.

The SP22 PVC Screen Head Adapter (37871) provides yet another screen option for the SP22 sampler. Using this adapter, a section of slotted 0.75-inch Schedule 40 PVC pipe may be lowered through the 2.25-inch probe rods using a string of fiush-threaded 0.75-inch Schedule 40 PVC Riser. An SP22 PVC Screen Plug (38968) is installed in the leading end of the slotted pipe prior to use. The slotted pipe may be cut and the screen plug installed to provide custom screen lengths .

An O-ring is located at the top of each stainless screen and on the screen adapters. When a screen is deployed, this O-ring maintains a seal between the top of the screen and the inner wall of the probe rods or expendable point holder as indicated in Figure 2.1. As a result, any liquid entering the tool string must first pass through the screen.

Screens are constructed such that equipment can be inserted into the screen cavity for sample collection as noted in the following section and illustrated in Figure 2.1, Step 4. This makes direct sampling possible from anywhere within the saturated zone.

The inner rod string and screen are generally removed prior to grouting through the 2.25-inch rod string as shown in Figure 2.1, Steps 5-6. However, a removable plug in the lower end of the screens allows for grouting through fiexible tubing extending out the bottom of the screen as with the Geoprobe® SP15/16 Groundwater Samplers if desired.

Sample Collection
Groundwater samples can be obtained from the SP22 screen in a number of ways. A common method utilizes 0.375-inch OD polyethylene (TB25L) or Tefion® (TB25T) tubing and a check valve assembly. The check valve (with check ball) is attached to one end of the tubing and inserted down the casing until it is immersed in groundwater. Water is then pumped through the tubing and to the ground surface by oscillating the tubing up and down.

An SP22 Check Valve Assembly (37893) is recommended if sampling through 1.25-inch light-weight center rods. The SP22 Check Valve Assembly is approximately 20 inches long to enable it to pass through the stepped diameters at each rod joint that may cause problems for other, shorter check valves.

An alternative means of collecting groundwater samples is to attach a peristaltic or vacuum pump to tubing that is inserted through the inner rods to within the SP22 screen. This method is limited in that water can be pumped to the surface from a maximum depth of approximately 26 feet (8 m). Another technique for groundwater sampling is to use a stainless steel Mini-Bailer Assembly (GW41). The mini-bailer is lowered down the inside of the casing below the water level where it fills with water and is then retrieved from the casing.

The latest option for collecting groundwater from the SP22 Sampler is to utilize a Geoprobe® MB470 Series Mechanical Bladder Pump (MBP)*. The MBP may be used to meet requirements of the low-fiow sampling protocol (Puls and Barcelona 1996, ASTM 2003). Through participation in a U.S. EPA Environmental Technology Verification study, it was confirmed that the MB470 can provide representative samples (EPA 2003).

**The Mechanical Bladder Pump is manufactured under U.S. Patent No. 6,877,965 issued April 12, 2005.*

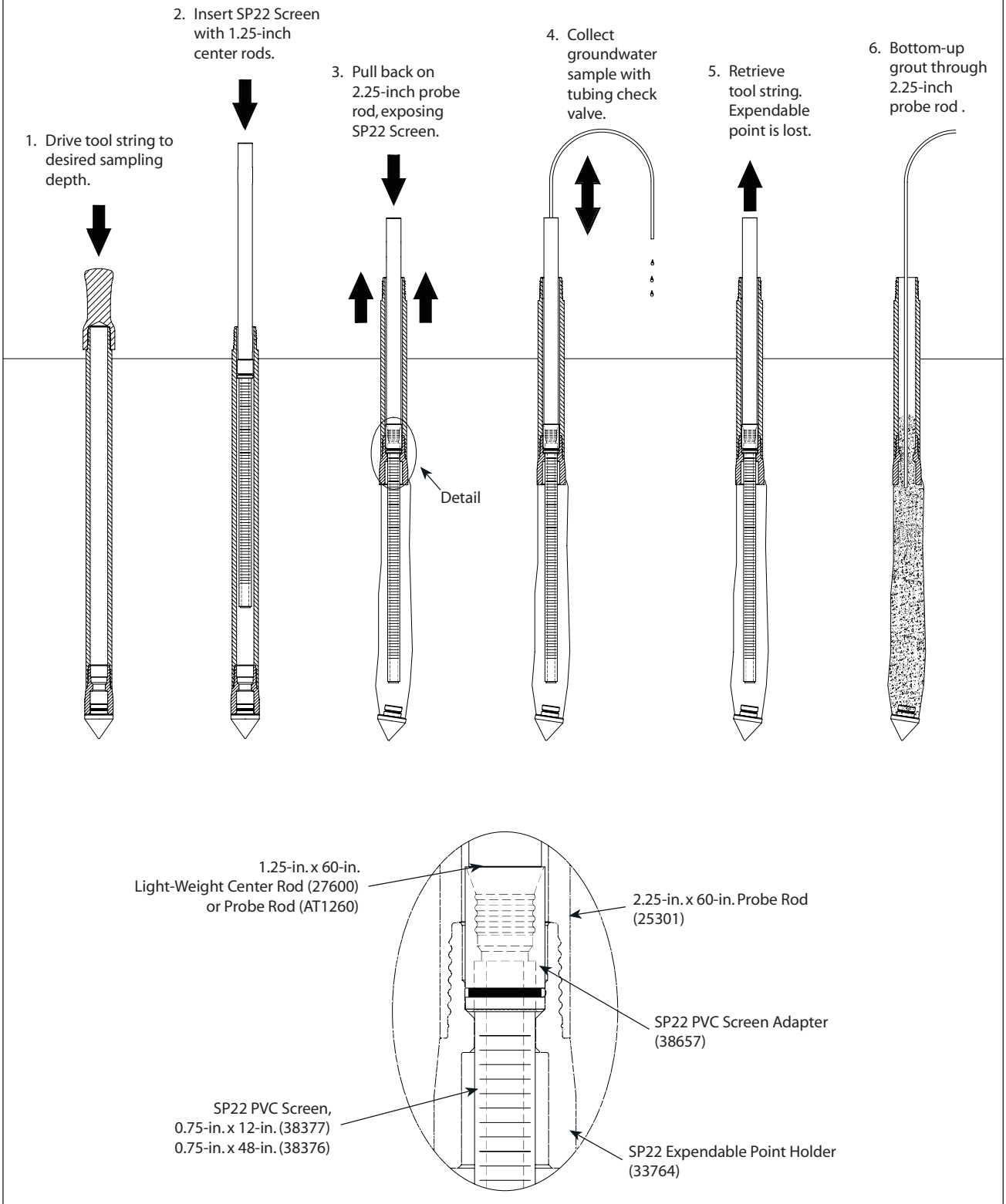
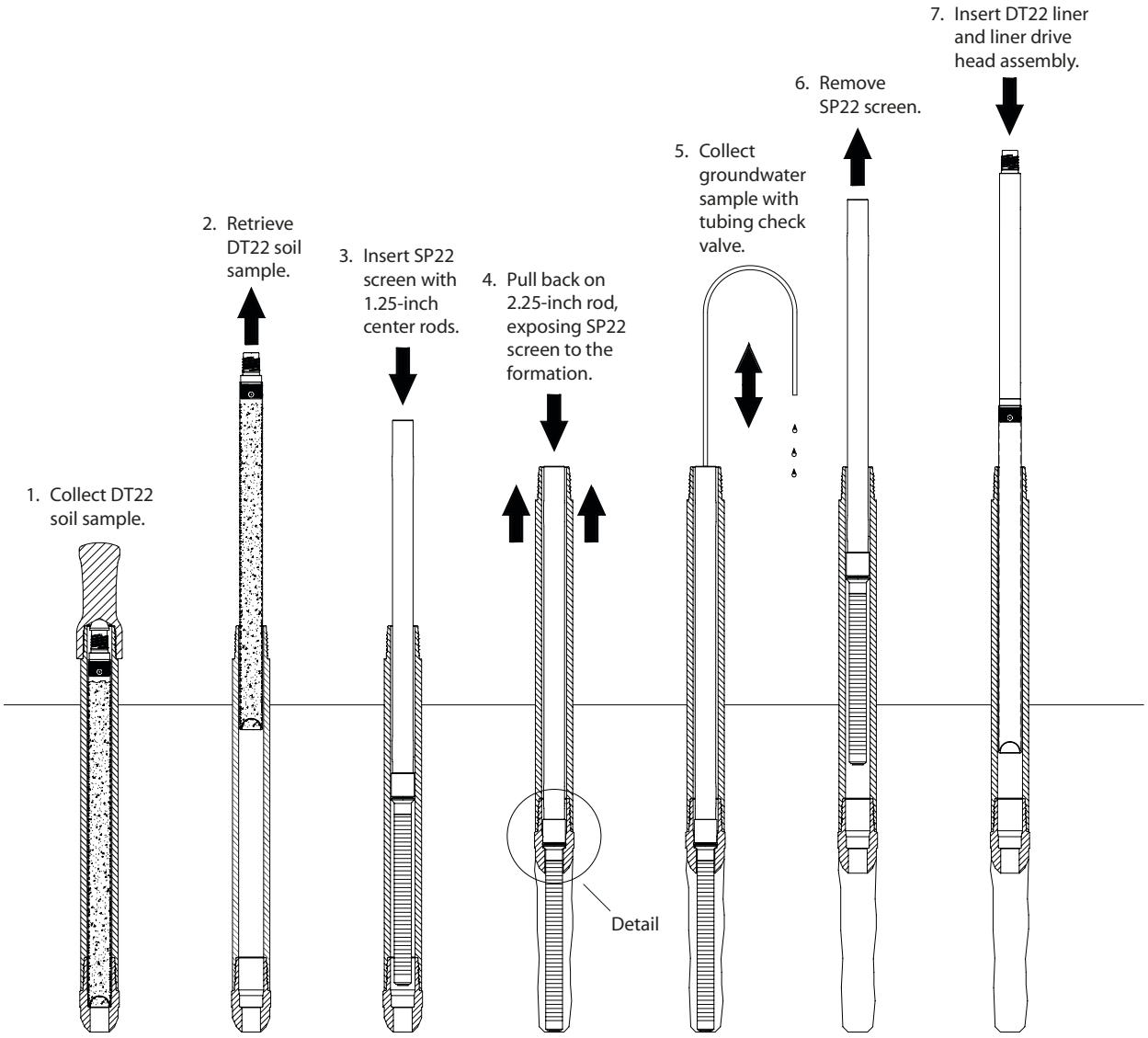


FIGURE 2.1
Screen Point 22 Groundwater Sampler - 0.75-inch PVC Screen Option



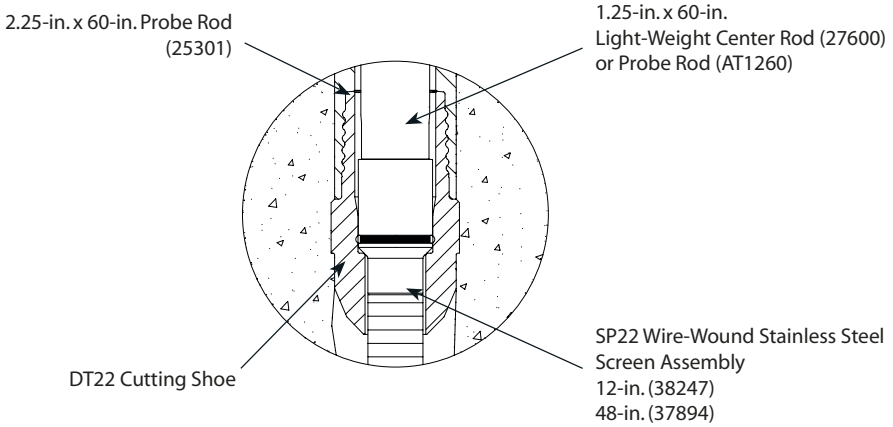


FIGURE 2.2
Screen Point 22 Groundwater Sampler Operation with DT22 Sampling System

3.0 TOOLS AND EQUIPMENT

The following tools and equipment can be used to successfully recover representative groundwater samples with the Geoprobe® Screen Point 22 Groundwater Sampler. Refer to Figures 3.1 and 3.2 for identification of the specified parts. Tools are listed below for the most common SP22 / 2.25-inch probe rod configurations. Additional rod sizes and accessories are available. Contact Geoprobe Systems® for information regarding tools and equipment options.

SP22 Sampler Parts	**Part Number**
SP22 Screen, Wire-Wound Stainless Steel, 4-Slot (48-in.)	37894
SP22 Screen, Wire-Wound Stainless Steel, 4-Slot (12-in.)	38247
Grout Plugs, PE (Pkg. of 25)	GW1552K
SP22 Screen, PVC, 10-Slot, 0.75-in. x 48-in	38376
SP22 Screen, PVC, 10-Slot, 0.75-in. x 48-inch, Kit (includes 2 each of 38376 and 38429)	*38664*
SP22 Screen, PVC, 10-Slot, 0.75-in. x 12-in	38377
SP22 Screen, PVC, 10-Slot 0.75-in. x 12-in., Kit (includes 2 each of 38377 and 38429)	*38667*
SP22 PVC Screen Plug	38968
SP22 PVC Screen Plug Kit (includes 10 of 38968)	*38530*
SP22 PVC Screen Adapter, 0.75-in. PVC x 1.25-in. Probe Rod Box	38657
SP22 PVC Screen Head Adapter, 0.75-in. (for fiush-threaded 0.75-in. Schedule 40 PVC)	37871
SP22 O-ring Kit (Pkg. of 10 O-rings for SP22 PVC screen adapters and stainless steel screens)	37853
O-rings, 0.75-in. PVC Riser (Pkg. of 25)	GW4401R
SP22 Expendable Point Holder, 2.25-in. Probe Rods, AT2045K and 19442 Points	33764
SP22 Expendable Point Holder, 2.25-in. Probe Rods, GW1555 Points*	43128

Outer Casing (2.125-inch Probe Rods) and Inner Rod String	**Part Number**
Probe Rod, 2.25-in. x 60-in.	25301
Expendable Drive Points, Steel, 2.45-in. OD (Pkg. of 25)	AT2015K
Expendable Drive Points, Steel, 2.45-in. OD, extended shank	19442
Expendable Points, steel, 1.625-in. OD (Pkg. of 25)*	GW1555K
Drive Cap, 2.25-in. Probe Rods, Threadless, (for GH60 and GH80 Series Hammers)	31530
O-Rings, 2.25-in. Probe Rods (Pkg. of 25)	AT2100R
Rod Grip Handle, 2.25-in. Probe Rods, (for GH60 and GH80 Series Hammers)	29385
Light-Weight Center Rod, 1.25-in. x 60-in.	27600
Probe Rod, 1.25-in. x 60-in.	AT1260
O-ring, 1.25-in. rods (Pkg. of 25)	AT1250R
Rod Grip Handle, 1.25/1.5-in. Rods, (for GH60 and GH80 Series Hammers)	15554
PVC Riser, 0.75-in. Schedule 40 x 60-inch	11747
PVC Pipe, 0.75-in. Schedule 40 x 60-inch, 10-Slot	17474

Grout Accessories	**Part Number**
High-Pressure Nylon Tubing, 0.375-in. OD / 0.25-in. ID, 100-ft. (30 m)	11633
Grout Machine, Auxiliary-Powered	GS2200
Grout System Accessories Package, 2.25-in. rods	GS1015

Groundwater Purging and Sampling Accessories	**Part Number**
Polyethylene Tubing, 0.375-in. OD, 500 ft	TB25L
Check Valve Assembly, 0.375-in. OD Tubing x 20 in. Long	37893
Water Level Meter, 0.438-in. OD Probe, 100 ft. cable	GW2000
Mechanical Bladder Pump**	MB470
Mini Bailer Assembly, Stainless Steel	GW41

* Not for use with GH80 Series Hydraulic Hammers

** Refer to the Standard Operating Procedure (SOP) for the Mechanical Bladder Pump (Technical Bulletin No. MK3013) for additional tooling needs.

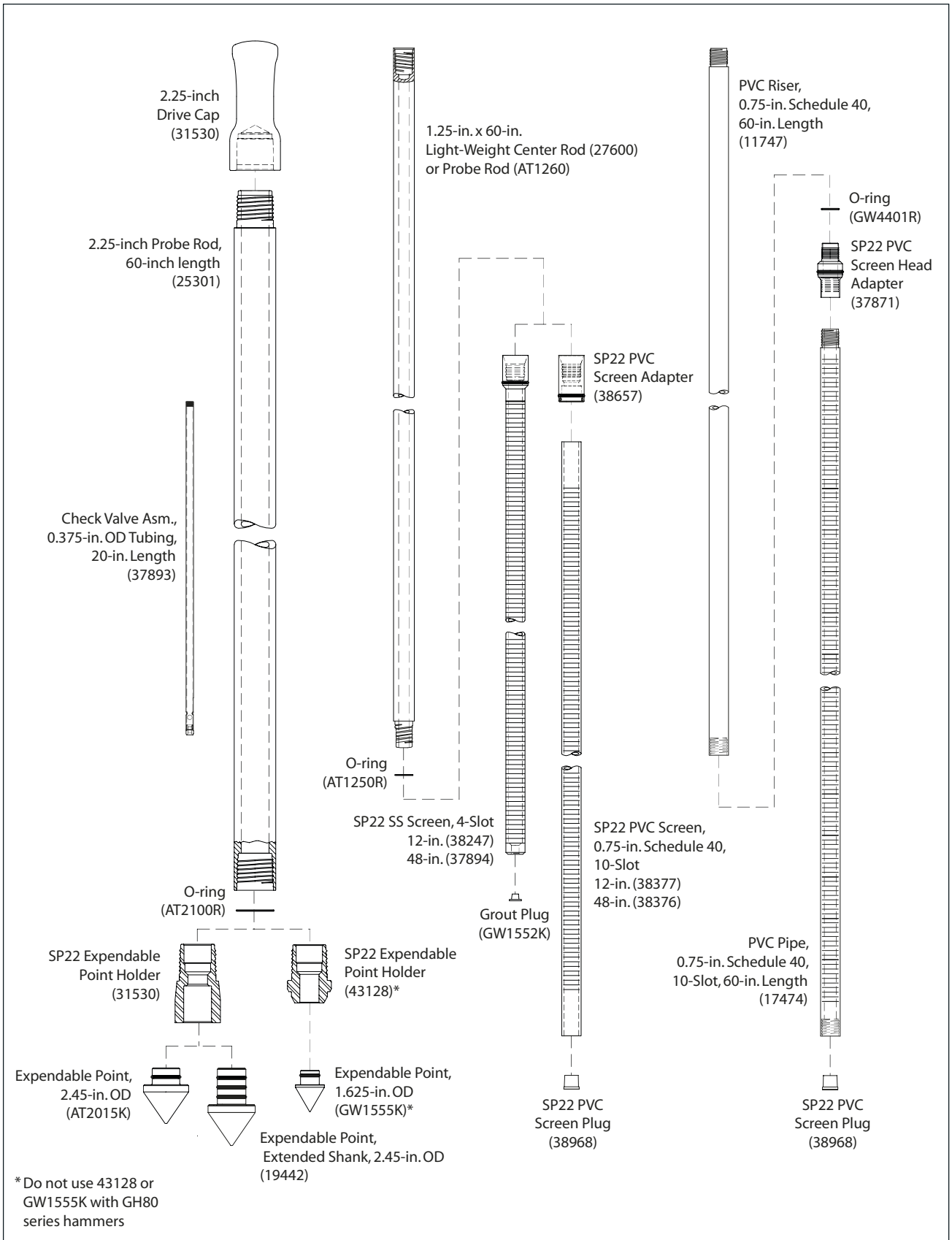


FIGURE 3.1
SP22 Sampler Parts

4.0 OPERATION

4.1 Basic Operation

The SP22 Sampler utilizes a stainless steel or PVC screen which is lowered (post-run) through an alloy steel 2.25-inch OD probe rod tool string. An expendable drive point is placed in an expendable point holder on the leading 2.25-inch probe rod prior to advancement (Fig. 4.1). This expendable point is removed and stays in the subsurface as the rods are pulled back to exposes the SP22 screen. O-rings on the probe rods , the expendable point holder, and the expendable drive point provide a watertight tool string which keeps contaminants out of the system as the 2.25-inch rods are driven to depth in preparation for installation of the SP22 screen.

Once the sampling interval is reached with the 2.25-inch probe rods, the stainless steel or PVC screen is lowered through the rods using 1.25-inch probe rods, 1.25-inch light-weight center rods, or 0.75-inch PVC riser pipe. The 2.25-inch tool string is then retracted while the screen is held in place with the inner rods or riser. The system is now ready for groundwater sampling. When sampling is complete, the inner rods and screen are removed for grouting during retrieval or the 2.25-inch rods. Alternatively, a removable plug is located in the bottom of the screens to allow grouting directly through the inner tool string with high-pressure tubing during retrieval.

4.2 Decontamination

In order to collect representative groundwater samples, all sampler parts must be thoroughly cleaned before and after each use. Scrub all metal parts using a stiff brush and a nonphosphate soap solution. Steam cleaning may be substituted for hand-washing if available. Rinse with distilled water and allow to air-dry before assembly.

4.3 Lead Rod Assembly (Fig. 4.1)

1. Place an O-ring on the expendable point holder.
2. Thread expendable point holder into the 2.25-inch probe rod.
3. Place an O-ring on a steel expendable drive point.
4. Firmly seat the expendable point in the expendable point holder.
5. Place 2.25-inch Drive Cap (31530) on the top of the 2.25-inch probe rod. The lead rod assembly is now ready to be driven to depth.

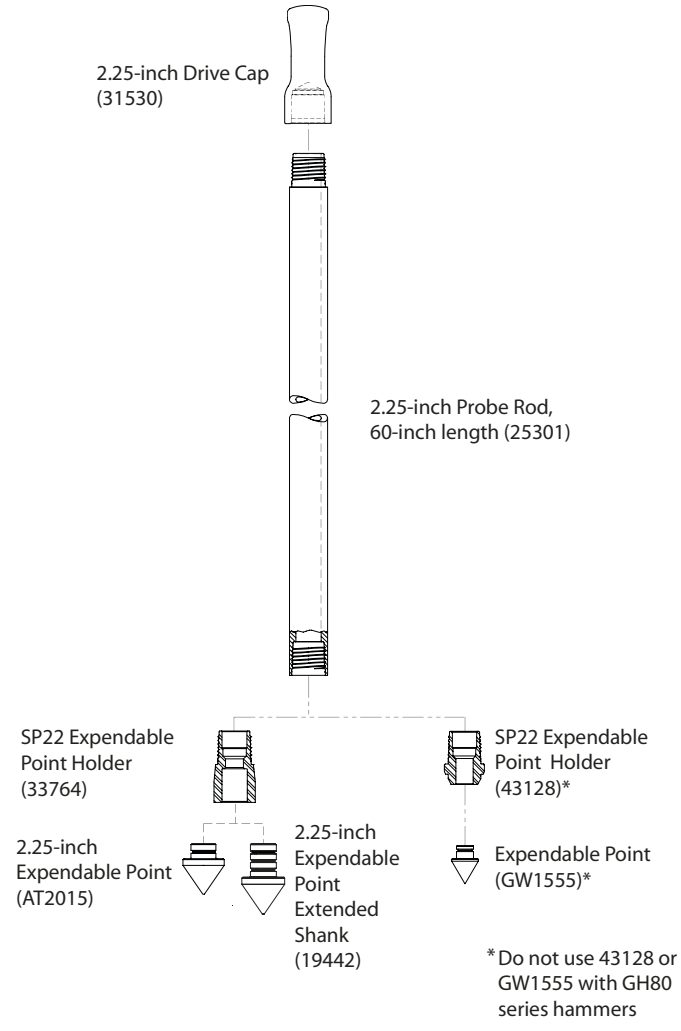


FIGURE 4.1
Lead Rod Assembly for the SP22 Groundwater Sampler

4.4 Advancing the Tool String (Fig. 4.2, step 1)

To provide adequate room for screen deployment with the Rod Grip Pull System, the probe derrick should be extended a little over halfway out of the carrier vehicle when positioning for operation.

1. Drive first 2.25-inch probe rod (as assembled in section 4.3).

2. Advance the tool string at a slow speed for the first few feet to ensure that the string is aligned properly.

3. Completely raise the hammer assembly. Remove the drive cap and place an O-ring in the top groove of the driven probe rod. Distilled water may be used to lubricate the O-ring if needed.

Add a probe rod (length to be determined by operator) and reattach the drive cap to the rod string. Drive the tool string the entire length of the new rod.

4. Repeat Step 3 until the desired sampling interval is reached. Approximately 12 inches (305 mm) of the last probe rod must extend above the ground surface to allow attachment of the puller assembly. A 12-inch (305 mm) rod may be added if the tool string is over-driven.

5. Remove the drive cap and retract the probe derrick away from the tool string.

4.5 Screen Deployment (Fig 4.2, step 2 - 4)

1. Attach an SP22 stainless steel or PVC screen to a 1.25-inch probe rod, 1.25-inch light-weight center rod, or 0.75-inch fiush-thread PVC riser using an SP22 PVC Screen Adapter (38657) or SP22 PVC Screen Head Adapter (37871) as shown in Figure 3.1. Note that the 38657 screen adapter is connected to the SP22 PVC screen using the setscrews provided with the adapter.

and lower it into the driven casing.

2. Lower the screen into the 2.25-inch probe rod casing and add rods or riser until the screen head contacts the bottom of the tool string.

3. Ensure that at least 48 inches (1219 mm) of rods or riser protrudes from the top 2.25-inch probe rod.

4. Maneuver the probe assembly into position for pulling.

5. Raise (pull) the outer 2.25-inch tool string while physically holding the screen in place with the inner 1.25-inch rods or 0.75-inch riser. A slight knock with the inner tool string will help to dislodge the expendable point and start the screen moving inside the probe rod.

Raise the hammer and outer tool string to expose the desired length of screen. The inner rods will begin raising with the outer rods when the screen adapter contacts the necked portion of the expendable point holder or DT22 Cutting Shoe. Use care when deploying a PVC screen so as not to break the screen when it contacts the expendable point.

6. Remove the rod grip handle, lower the hammer assembly, and retract the probe derrick. Remove the top 2.25-inch probe rod.

7. Groundwater samples can now be collected with a mini-bailer, peristaltic or vacuum pump, tubing bottom check valve assembly, bladder pump, or other acceptable small diameter sampling device.

When inserting tubing or a bladder pump down the rod string, ensure that it enters the screen interval. The leading end of the tubing or bladder pump will sometimes catch at the screen head giving the illusion that the bottom of the screen has been reached. An up-and-down motion combined with rotation helps move the tubing or bladder pump past the lip and into the screen.

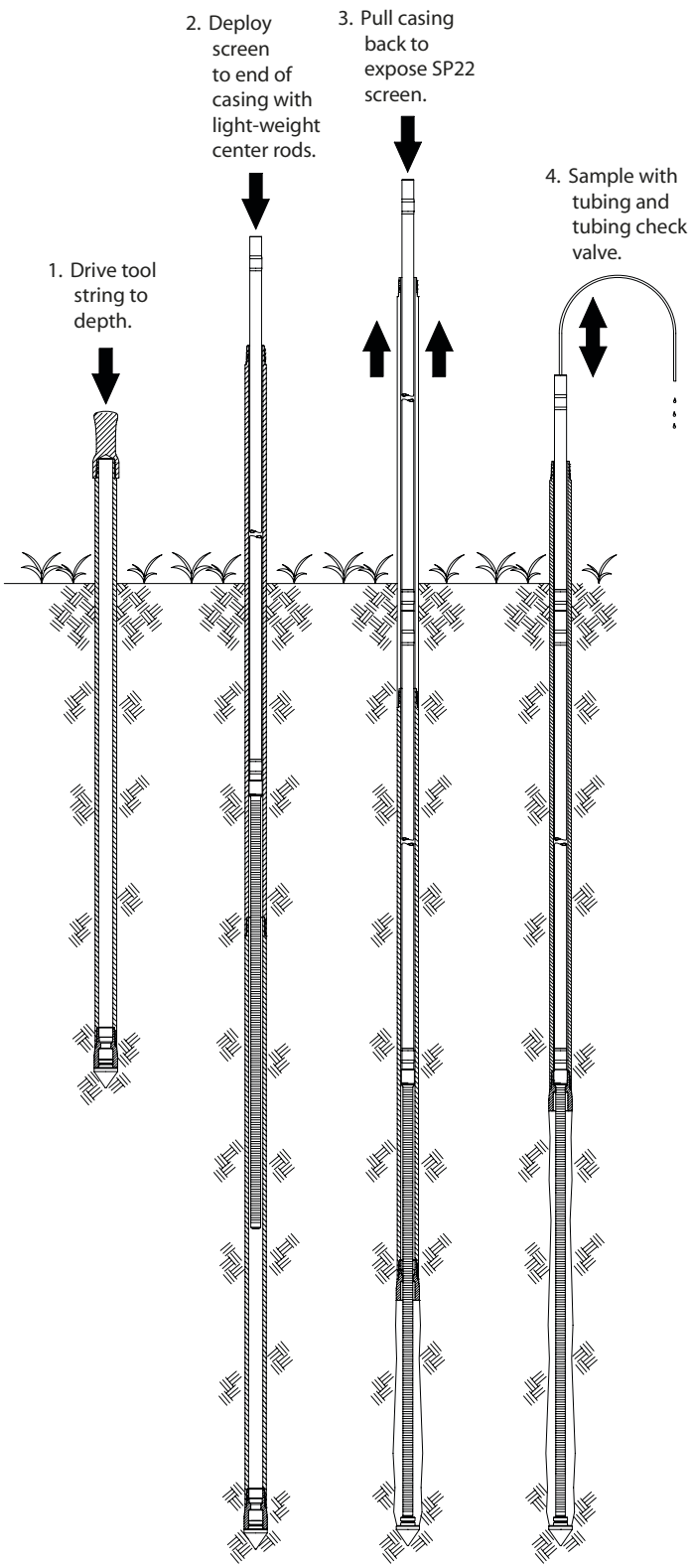


FIGURE 4.2
Screen Deployment for SP22 Sampler

4.6 Abandonment Grouting for SP22 Screens

The SP22 Sampler can meet ASTM D 5299 requirements for abandoning environmental wells or borings when grouting is conducted properly. A removable grout plug makes it possible to deploy tubing through the bottom of the SP22 screens, but the easiest method is to remove the inner string of rods; including the SP22 screen. A Grout Machine is then used to pump grout into the open probe hole as the outer casing is withdrawn. The following procedure is presented as an example only and should be modified to satisfy local abandonment grouting regulations. (Figure 4.3)

1. Maneuver the probe assembly into position for pulling.

2. High-Pressure Nylon Tubing (11633) is inserted down through the probe rods through the bottom of the expendable point holder (Fig. 4.3).

   Note: *All probe rods remain strung on the tubing as the tool string is pulled. Provide extra tubing length to allow sufficient room to lay the rods on the ground as they are removed. An additional 20 feet is generally enough.*

3. Operate the grout pump while pulling the first rod with the rod grip pull system. Coordinate pumping and pulling rates so that grout fills the void left by the sampler. After pulling the first rod, release the rod grip handle, fully lower the hammer, and regrip the tool string. Unthread the top probe and slide it over the tubing placing it on the ground near the end of the tubing.

4. Repeat Step 5 until the tool string is retrieved. Do not bend or kink the tubing when pulling and laying out the probe rods. Sharp bends create weak spots in the tubing which may burst when pumping grout. Remember to operate the grout pump only when pulling the rod string. The probe hole is thus filled with grout from the bottom up as the rods are extracted.

5. Promptly clean all probe rods and sampler parts before the grout sets up and clogs the equipment.

4.7 Retrieving the Screen Point 22 Sampler

If grouting is not required, the Screen Point 22 Sampler can be retrieved by pulling the probe rods as with most other Geoprobe® applications. The Rod Grip Pull System should be used for this process as it allows the operator to remove rods without completely releasing the tool string. This avoids having the probe rods fall back downhole when released during the pulling procedure. A standard Pull Cap (33622) may still be used if preferred. Refer to the Owner's Manual for your Geoprobe® direct push machine for specific instructions on pulling the tool string.

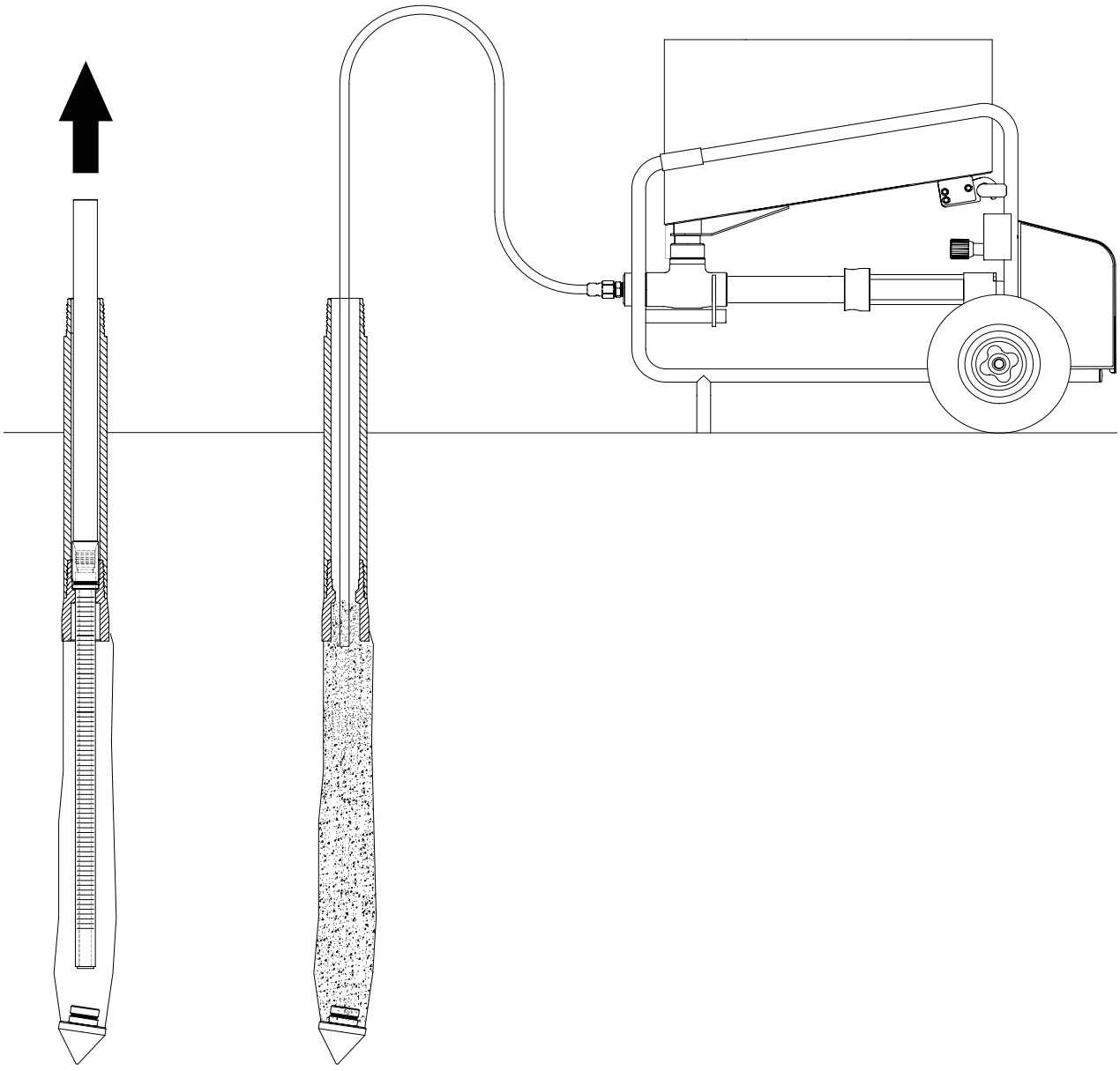


FIGURE 4.3
Abandonment Grouting for the SP22 Sampler

5.0 REFERENCES

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Equipment and tool specifications, including weights, dimensions, materials, and operating specifications included in this brochure are subject to change without notice. Where specifications are critical to your application, please consult Geoprobe Systems®.



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TGI - Equipment and Reagent Blank Sample Collection for PFAS Analysis

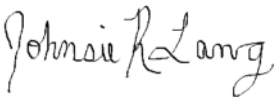
Rev: 2

Rev Date: February 21, 2022

Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
--	0	October 2, 2018	All	TGI – Equipment and Reagent Blank Sample Collection for PFAS Analysis	Erika Houtz
Outdated template	1	December 10, 2021	All	Updated to current TGI format. Also updated sections to match the most recent TGI for PFAS Field Sampling Guidance (Arcadis 2021)	Kevin Engle
Template Updates	2	February 21, 2022	All	Additional template updates	Kevin Engle, Johnsie Lang

Approval Signatures

Prepared by:		2/21/2022
	Kevin Engle, PG (WY)	Date
Reviewed by:		2/21/2022
	Johnsie Lang, PhD (Subject Matter Expert)	Date

1. Introduction

This document is intended to provide guidance to field staff collected equipment blanks for Per- and Polyfluoroalkyl Substances (PFAS). The content in this document describes the intended use, scope and application, personnel qualifications, equipment, cautions, health and safety considerations, procedures, waste management, data recording and management, and quality assurance of PFAS sampling.

2. Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3. Scope and Application

Equipment and reagent blanks will be collected in the field during sampling activities and submitted for laboratory analysis. These samples are primarily intended to verify that sampling and decontamination practices are effectively preventing cross-contamination caused by reusable sample equipment or other per- and polyfluoroalkyl substances (PFAS)-containing materials.

The intent of this Technical Guidance Instruction (TGI) is to provide instructions for collection of equipment and reagent blanks. More detailed instructions related to general PFAS sampling considerations is provided in the Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance (Arcadis 2021).

4. Personnel Qualifications

Equipment and reagent samples will be collected by persons who have been trained in proper sampling procedures under the guidance of an experienced field geologist, engineer, or technician. Blank sampling should be completed with a two-person sampling team.

5. Equipment List

The following equipment and materials must be available for equipment and reagent blank sampling:

- Site plan which specifies frequency/quantity of blank sampling;
- Relevant work plan (e.g., PQAPP);
- Site Safety and Health Plan (SSHP);
- Appropriate health and safety equipment, as specified in the SSHP;
- Laboratory-provided "PFAS-free" water;
- Nitrile gloves;
- Dedicated plastic sheeting (preferably low-density polyethylene) or other clean surface to prevent sample contact with the ground;
- Pail or bucket with closable lid for excess rinse water;
- Garbage bags;
- Appropriate sample containers and labels;
    - Labeled high density polypropylene (HDPE) sample bottles: see the Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance (Arcadis 2021) for PFAS-specific considerations;
    - Ziploc®-style bags to hold ice and samples;
    - Packing and shipping materials;
    - Chain-of-Custody (COC) Forms; see the Sample Chain of Custody Standard Operating Procedure (SOP) for reference (Arcadis 2017a);
    - Appropriate transport containers (coolers) with ice and appropriate labeling; no blue ice is to be used.
- Decontamination:
    - Equipment cleaning materials: see the Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance (Arcadis 2021) or the Groundwater and Soil Sampling Equipment Decontamination TGI (Arcadis 2017b) as applicable;
    - An organic solvent such as isopropanol, methanol, or acetone should be used to decontaminate reusable equipment if it can be brought to the site safely. While strongly recommended, the use of solvents may be excluded for project-specific health and safety concerns. Refer to Section 7.1.1 for more details.
    - Drum labels as required for investigation-derived waste handling: see the Investigation-Derived Waste Handling and Storage TGI for details (Arcadis 2017c);

- Field Notes:
  - Pens, pencils, and/or fine point Sharpies® for writing;
  - Appropriate field forms;
  - Clipboards, field binders, field notebook, and field note pages that are not waterproof.

6. Cautions

In general, sampling techniques used for PFAS sample collection are consistent with conventional sampling techniques used in the environmental industry, but special consideration is made regarding PFAS-containing materials and cross-contamination potential. The most important consideration during PFAS-related sampling is to prevent contact between sample media and suspect PFAS sources. During collection of equipment and reagent blanks, the sampled media (i.e., PFAS-free water) should not contact anything but the sample container. New nitrile gloves should be donned after handling of any non-dedicated sampling equipment; contact with contaminated surfaces; and whenever judged necessary by field personnel. When in doubt change your gloves. More detailed instructions related to general PFAS sampling considerations is provided in the TGI - Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance (Arcadis 2021).

7. Health and Safety Considerations

Field activities associated with equipment and reagent blank sampling will be performed in accordance with the SSHP, a copy of which will be present on site during such activities. Additional health and safety considerations can be found in TGI for PFAS Field Sampling Guidance (Arcadis 2021).

8. Procedure

The specific procedure for equipment and reagent blank sampling was developed after careful review and consideration of project data quality objectives. Procedures for equipment blank sampling and reagent blank sampling are further described in this section. Note: the laboratory has to analyze the entire sample bottle for aqueous solutions of PFASs. When collecting each blank, fill two sample bottles and instruct the lab to hold one of them in reserve. If an unacceptable detection occurs in a blank, the second bottle of sample may be analyzed.

For additional sampling considerations, reference the TGI for PFAS Field Sampling Guidance (Arcadis 2021).

8.1 Blank Sampling

8.1.1 Decontamination

Prior to collecting blank samples, the applicable piece of equipment should be properly decontaminated following these steps:

- Hand Tools and Sampling Devices (including hand augers and bladder pumps)
  1. Don new pair of nitrile gloves prior to decontamination
  2. Remove o-rings and bladder (applies only to bladder pump)

3. Scrub using a plastic brush and a non-phosphate soap free of volatile organic compounds (VOCs) (e.g., Liquinox, Alconox);
4. Double-rinse in deionized or distilled water;
5. Rinse once with the site-approved organic rinsing solvent (e.g., isopropanol, methanol, acetone);
6. Rinse once with PFAS-free water;
7. Collect all rinsate in a sealed pail for disposal;
8. Allowed time to air dry prior to re-use.
9. Insert new o-rings and bladder (applies only to bladder pump)

While strongly recommended, the use of solvents may be excluded for project-specific health and safety concerns. If solvents are prohibited, then additional procedures should be evaluated by the project team. Contingencies could include the use of dedicated sampling equipment at each sampling location or amending laboratory procedures to mitigate the increased risk of cross-contamination.

The following decontamination procedure could be utilized when organic solvent use is not possible:

1. Don new pair of nitrile gloves prior to decontamination
2. Remove o-rings and bladder (applies only to bladder pump)
3. Scrub using a plastic brush and a non-phosphate soap free of VOCs (e.g., Liquinox, Alconox);
4. Single-rinse in deionized or distilled water;
5. Scrub using a plastic brush and a non-phosphate soap free of VOCs (e.g., Liquinox, Alconox);
6. Rinse twice with deionized water and once with PFAS-free water;
7. Collect all rinsate in a sealed pail for disposal;
8. Allowed time to air dry prior to re-use.
9. Insert new o-rings and bladder (applies only to bladder pump)

- Drilling Rods
  - Drive casings and other drilling equipment will be steam cleaned or replaced with new equipment between boreholes.
  - The drilling equipment will be cleaned in an area designated by the supervising engineer or geologist that is located outside of the work zone.

After verifying the piece of equipment is properly decontaminated, and after determining an equipment blank is warranted per the sampling quality assurance / quality control (QA/QC) plan, follow the specific procedures for the relevant type of equipment found in the following sections.

8.1.2 Drilling Equipment (Hand Auger or Cutting Shoe/Drill Rod)

Two field personnel should participate in the collection of the equipment blank. One person (“Field Personnel #1”) should hold the sampling bottle and collect the sample, and the second person (“Field Personnel #2”) should pour the rinse water.

The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep bottle lid on until immediately prior to sample collection.
2. Lay down dedicated plastic sheeting or other clean surface to prevent sample contact with the ground.
3. Place the sealable bucket or pail on top of plastic sheeting.

4. Don a new pair of nitrile gloves prior to blank collection (Field Personnel #1 and #2). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.
5. Open the sample container and position the piece of clean, decontaminated sample equipment (i.e., hand auger or drilling rod/cutting shoe) above the container (Field Personnel #1). Keep the sample cap in the hand of the sampler (Field Personnel #1) until it is replaced on the bottle.
    a. The bucket of the hand auger can be removed from the rods/handle and held manually.
    b. The drillers should assist the field staff with removing the cutting shoe from the drill string and positioning it for sampling.
6. Slowly pour laboratory-provided "PFAS-free" water over any surface of the decontaminated sampling device that previously contacted sampled material (Field Personnel #2).
    a. Pour water through the inside of the hand auger bucket while manually rotating the bucket so that "PFAS-free" water contacts all sides of the sampling device. Collect runoff in the sample container (Field Personnel #1), making sure that any excess "PFAS-free" water is contained in the sealable bucket or pail.
    b. Pour water through the inside of cutting shoe while drilling contractor holds and manually rotates the shoe so that "PFAS-free" water contacts all sides of the shoe (Field Personnel #2). Collect runoff in the sample container, making sure that any excess "PFAS-free" water is contained in the sealable bucket or pail.
7. After collecting the necessary sample volume, place cap back on the sample bottle (Field Personnel #1). The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
8. Collect the second bottle with the same procedure (Steps 5 to 7), if collecting a backup.
9. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.
10. Add sample to the laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
11. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
12. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.3 Reusable Sediment Sampling Equipment (Stainless-Steel Hand Tools, Petite Ponar Grab Sampler)

Two field personnel should participate in the collection of the equipment blank. One person ("Field Personnel #1") should hold the sampling bottle and collect the sample, and the second person ("Field Personnel #2") should pour the rinse water.

The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep bottle lid on until immediately prior to sample collection.
2. Lay down dedicated plastic sheeting or other clean surface to prevent sample contact with the ground.
3. Place sealable bucket or pail on top of plastic sheeting.
4. Don new pair of nitrile gloves prior to blank collection (both field personnel). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.

5. Open sample container and position the clean, decontaminated piece of sample equipment (i.e., hand tool, grab sampler) above the container (Field Personnel #1). Keep the sample cap in the hand of the sampler (Field Personnel #1) until it is replaced on the bottle.
6. Slowly pour the laboratory-provided "PFAS-free" water over any surface of the sampling device that contacted sampled material (Field Personnel #2).
    a. Pour water over front and back of all decontaminated hand tools such as spoons, spatulas, and trowels so that "PFAS-free" water touches all sides of the sampling device. Collect runoff in the sample container, making sure that any excess "PFAS-free" water is contained in the sealable bucket or pail (Field Personnel #1).
    b. Pour water through inside of the decontaminated Petite Ponar Grab Sampler while rotating the sampler (or the "PFAS-free" water container) so that "PFAS-free" water contacts all interior sides of the sampler (Field Personnel #2). Collect runoff in the sample container, making sure that any excess "PFAS-free" water is contained in the sealable bucket or pail.
7. After collecting the necessary sample volume, place cap back on the sample bottle (Field Personnel #1). The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
8. Collect the second bottle with the same procedure (Steps 5 to 7), if collecting a backup.
9. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.
10. Add the sample to the laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
11. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
12. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.4 Disposable Sediment Sampling Equipment (Lexan TM Liner Sleeve)

Two field personnel should participate in the collection of the equipment blank. One person ("Field Personnel #1") should hold the sampling bottle and collect the sample, and the second person ("Field Personnel #2") should pour the rinse water.

The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep the bottle lid on until immediately prior to sample collection.
2. Lay down dedicated plastic sheeting or other clean surface to prevent sample contact with the ground.
3. Place sealable bucket or pail on top of plastic sheeting.
4. Don new pair of nitrile gloves prior to blank collection (both field personnel). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.
5. Open sample container and position a clean, new, and unused section of LexanTM liner above the container (Field Personnel #1). Keep the sample cap in the hand of the sampler (Field Personnel #1) until it is replaced on the bottle.
6. Slowly pour laboratory-provided "PFAS-free" water over any surface of the sampling device that contacted sampled material (Field Personnel #2).

   a. Pour water through inside of Lexan TM liner while rotating the liner so that “PFAS-free” water contacts all interior sides of the liner. Collect runoff in the sample container, making sure that any excess “PFAS-free” water is contained in the sealable bucket or pail.
7. After collecting the necessary sample volume, place cap back on the sample bottle (Field Personnel #1). The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
8. Collect the second bottle with the same procedure (Steps 5 to 7), if collecting a backup.
9. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.
10. Add sample to the laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
11. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
12. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.5 Reusable Water Sampling Equipment (Peristaltic Pump, Bladder Pump, Stainless-Steel Bailer)

8.1.5.1 Peristaltic Pump

Two field personnel should participate in the collection of the equipment blank. One person (“Field Personnel #1”) should hold the sampling bottle and collect the sample, and the second person (“Field Personnel #2”) should set up the pump and pour/ transfer the blank water.

The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep bottle lid on until immediately prior to sample collection.
2. Lay down dedicated plastic sheeting other clean surface to prevent sample contact with the ground.
3. Don new pair of nitrile gloved prior to blank collection (both field personnel). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.
4. Pour laboratory-supplied “PFAS-free” water into a clean HDPE sample bottle (Field Personnel #2).
5. Insert new HDPE tubing into the HDPE bottle containing “PFAS-free” water and connect tubing to peristaltic pump (with new silicone tubing) (Field Personnel #2).
6. Open sample container, keeping the sample cap in the hand of the sampler until it is replaced on the bottle (Field Personnel #1).
7. Turn the peristaltic pump on and slowly pump the “PFAS-free” water into the labeled sample container (Field Personnel #2).
8. After collecting the necessary sample volume, place cap back on the sample bottle (Field Personnel #1). The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
9. Collect the second bottle with the same procedure (Steps 6 to 8), if collecting a backup.
10. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.

11. Add the sample to the laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
12. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
13. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.5.1 Bladder Pump

1. Two field personnel should participate in the collection of the equipment blank. One person ("Field Personnel #1") should hold the sampling bottle and collect the sample, and the second person ("Field Personnel #2") should set up the pump and pour/ transfer the blank water.
2. The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.
3. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep the bottle lid on until immediately prior to sample collection.
4. Lay down dedicated plastic sheeting or other clean surface to prevent sample contact with the ground.
5. Don new pair of nitrile gloved prior to blank collection (both field personnel). Do not use gloved hands to handle papers, pens, clothes, equipment, etc., before collecting samples.
6. Pour laboratory-supplied "PFAS-free" water into an approved container (to avoid PFAS cross-contamination) large enough to submerge the bladder pump.
7. After properly decontaminating the bladder pump and replacing the bladder, attach a new section of HDPE tubing to the bladder pump, long enough to hold and direct flow into the labeled sample container. Submerge the bladder pump into the approved container of "PFAS-free" water (Field Personnel #2).
8. Open sample container, keeping the sample cap in the hand of the sampler until it is replaced on the bottle (Field Personnel #1).
9. Turn the bladder pump on and slowly pump the "PFAS-free" water into the labeled sample container (Field Personnel #2).
10. After collecting the necessary sample volume, place cap back on the sample bottle. The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
11. Collect the second bottle with the same procedure (Steps 6 to 8), if collecting a backup.
12. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.
13. Add sample to laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
14. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
15. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.5.2 Stainless-Steel Bailer

Two field personnel should participate in the collection of the equipment blank. One person ("Field Personnel #1") should hold the sampling bottle and collect the sample, and the second person ("Field Personnel #2") should pour the rinse water.

The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep bottle lid on until immediately prior to sample collection.
2. Lay down dedicated plastic sheeting or other clean surface to prevent sample contact with the ground.
3. Place sealable bucket or pail on top of plastic sheeting.
4. Don new pair of nitrile gloves prior to blank collection (both field personnel). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.
5. Open sample container (Field Personnel #1) and position the bailer above the container (Field Personnel #2). Keep the sample cap in the hand of the sampler (Field Personnel #1) until it is replaced on the bottle.
6. Fill the stainless-steel bailer with enough laboratory-provided “PFAS-free” water to collect the necessary sample volume (Field Personnel #2).
7. Slowly pour laboratory-provided “PFAS-free” water from the stainless-steel bailer into the sample container (Field Personnel #2).
8. After collecting the necessary sample volume, place cap back on the sample bottle. The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
9. Collect the second bottle with the same procedure (Steps 5 to 8), if collecting a backup.
10. Place filled sample bottles in sealed Ziploc® bags, record any label information that was not pre-filled out, if necessary (e.g., sample time). Record the label information and time of sampling in the field notes and sampling forms.
11. Fill out the laboratory COC and check against the labels on the Equipment Blank sample bottle(s) progressively after each Equipment Blank is collected. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
12. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in a separate cooler from any other types of samples.
13. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.6 Field Reagent Blank Sampling

Two field personnel should participate in the collection of the reagent blank. One person (“Field Personnel #1”) should hold the sampling bottle and collect the sample, and the second person (“Field Personnel #2”) should pour the blank water.

This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep the bottle lid on until immediately prior to sample collection.
2. Don new pair of nitrile gloves prior to blank collection (both field personnel). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.
3. Open sample container, keeping the sample cap in the hand of the sampler (Field Personnel #1) until it is replaced on the bottle (Field Personnel #1).
4. Slowly pour laboratory-provided “PFAS-free” water from the laboratory-provided container into the sample container (“Field Personnel #2”).
5. After collecting the necessary sample volume, place cap back on the sample bottle. The bottle should be filled approximately full, but some headspace in the bottle is acceptable.

6. Collect the second bottle with the same procedure (Steps 3 to 5) if collecting a backup.
7. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.
8. Add sample to the laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
9. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
10. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

9. Waste Management

Excess water generated during equipment and reagent blank collection procedures will be collected and contained on site in appropriate containers, (see the Investigation-Derived Waste Handling and Storage TGI for details [Arcadis 2017c]). All investigation-derived waste (IDW) generated will be placed in Department of Transportation approved containers, sealed, and labeled. Containerized IDW will be stored on site until it is profiled and subsequently transported to an approved facility for disposal or recycling. Waste manifests for all IDW suspected to have come into contact with PFAS should clearly note the presence of PFAS. Additional IDW sampling and management details will be provided in the site-specific Work Plan (QAPP addendum) and will be consistent with applicable client policies and requirements. Disposable personal protective equipment (e.g., gloves, disposable clothing, disposable equipment) will be placed in plastic bags. These bags will be transferred into appropriately labeled containers for appropriate disposal.

10. Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

If digital data collection isn't possible, waterproof field books should be avoided for field notes. Instead, field notes on loose paper on Masonite, plastic, or aluminum clip boards is preferred. Please note that newer Rite in the Rain® notebooks are approved for PFAS sampling. Other requirements for field notes include:

- Pens, pencils, and fine point Sharpies® may be used.
- Keep field notes and writing implements away from samples and sampling materials.
- Do not write on sampling bottle labels unless the sample bottle covers are tightly closed.
- Complete sampling logs in their entirety.

- Make sure COC forms are properly completed. Verify that the analysis method requested is US EPA Method 537.1 for potable water and includes the appropriate analytes desired for analysis.

11. Quality Assurance

Refer to quality control requirements for the project to ensure that appropriate QA/QC samples are collected. When collecting QA/QC samples, the same guidelines apply as when collecting regular samples – specifically:

- **Duplicate samples of each equipment blank and reagent blank should be collected and submitted to the laboratory with instructions to hold for analysis. The purpose of this sample is to provide analytical back-up in case there are any issues with the original blank sample.**
- Samples should be collected in laboratory-supplied HDPE bottles;
- Bottle caps must remain in the hand of the sampler until replaced on the bottle;
- Labels must be completed after the caps have been placed back on each bottle; and,

Samples must be stored in appropriate transport containers (coolers) with ice (Ziploc®-type bags for use as ice containers) with appropriate labeling. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**

12. References

Arcadis. 2021. TGI – Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance. Rev. 9. October 22.

Arcadis. 2017a. SOP – Sample Chain of Custody, Rev. #1. May 23.

Arcadis. 2017b. TGI – Groundwater and Soil Sampling Equipment Decontamination, Rev. #0. February 23.

Arcadis. 2017c. TGI – Investigation-Derived Waste Handling and Storage, Rev. #0. February 23.

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TGI – Per- and Polyfluoroalkyl Substances (PFAS) Field Sampling Guide

Rev: 12

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Version Control

Revision No.	Date Issued	Page No.	Description	Reviewed By
0	April 27, 2017	All	Initial Release	Erica Kalve Erika Houtz Sue Tauro
1	June 19, 2018	1 through 4 and 17	Updated Information on Sampling Materials	Erica Kalve Erika Houtz
2	October 15, 2018	6 to 16	Minor updates on laboratory elements, updates to decontamination procedures, and clarification on equipment and reagent blank collection	Erika Houtz Erica Kalve
3	December 17, 2018	4, 6, 17	Removed Sharpies from acceptable field writing implements; Changed language in Section 3.2 and Section 10.5 to provide stricter guidance for DoD projects.	Erika Houtz, Erica Kalve
4	March 26, 2019	4,5	Removed Citranox from acceptable Decon solutions in Table 1a, added all fluoropolymer containing materials to prohibited items in Table 1b. Made a correction that Liquinox contains trace levels of 1,4 Dioxane, not Alconox.	Erika Houtz
5	October 16, 2020	14	Added Air Force preference to sample surface water at surface for Air Force investigations.	Erika Houtz
6	March 23, 2021	4, 5, 7, 12, 13, 14, 15, 16, 17	Made clarifications that fine/ultra-fine point Sharpies are allowed. Referenced 2018 MDEQ sampling guidance. Made updates to 'After Sample Collection' in Section 7.	Kevin Engle

7	April 18, 2021	All	Changed title from Poly- and Perfluoroalkyl Substances to **"Per- and Polyfluoroalkyl Substances"** and changed PFASs to PFAS.	Rosario Varrella, Erika Houtz
8	May 4, 2021	12, 13, 15, 16	Clarified that sample containers should have an HDPE lined screw cap and that LDPE plastic sheeting should be used.	Kevin Engle, Erika Houtz
9	October 20, 2021	Note that numbers have shifted one page forward relative to prior versions. 5, 7, 9-12, 15, 16, 18-25.	Specific acceptable sunscreen and insect repellent brands were added to Table 1. Clarified language regarding footwear and H&S trainings. Laboratories section and Section 10.5 was updated to reflect new laboratory names and an updated version of the QSM. Sections 5 and 6 were updated to provide clearer language on health and safety protocols for sunscreen, insect repellent, and rain events. Added language to specify decontamination of reusable equipment prior to initial use in Section 7.1. Section 8 on Waste Management was updated to state that waste storage and disposal should be determined in the site specific workplan. Section 9 was updated to include Rite in the Rain® notebooks as approved for PFAS sampling. Changed the term "sample port" to "sample location" when describing where to place plastic sheeting. Section 10.1 was updated to indicate an equipment blank can be collected for unvetted hazard controls that contact a sample. References were updated to include the newer version of the DoD QSM, MDEQ Sampling Guidance, and	Kevin Engle, Erika Houtz

			California State Water Board PFAS Sampling Guidance.	
10	January 26, 2022	Various, Section 7	TGI formatted to comply with new QMS TGI template and Arcadis brand compliance. Indicated to avoid use of anti-fog spray on safety glasses due to possible presence of PFAS.	Rosario Varrella, Kevin Engle
11	January 13, 2023	Various	TGI updated to include specific information found in the June 2022 ITRC Guidance document. Updates reflecting current state of the practice analytical procedures. Table 1a and Table 1b updates	Kevin Engle, Joey Matzke, Lauren March
12	September 20, 2023	Various	Decontamination procedure in Section 8.1 has been updated to remove solvent rinse. Procedure is now: Scrub with alconox, rinse with DI/distilled water, rinse with “PFAS-free” water, repeat. Updated “Shipping” section 8.4, allowing for Saturday delivery with laboratory approval. Made various minor corrections and clarifications related to specific brand names	Alexander Hamilton, Johnsie Lang

Approval Signatures

Prepared by:		9/20/2023
	Alexander Hamilton, PG Geologist (Preparer)	Date
Reviewed by:		9/20/2023
	Johnsie Lang, PhD (Subject Matter Expert)	Date

1 Introduction

This document is intended to provide guidance to field staff sampling for Per- and Polyfluoroalkyl Substances (PFAS). The content in this document describes the intended use, scope and application, personnel qualifications, equipment, cautions, health and safety considerations, procedures, waste management, data recording and management, and quality assurance of PFAS sampling.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

The purpose of this Technical Guidance Instructions (TGI) is to provide guidance on field sampling to be used for **Per- and Polyfluoroalkyl Substances** (PFAS). This protocol was adapted from various sources including Arcadis Australia, Transport Canada, and the U.S Army Corp of Engineers (USACE) Omaha. In general, sampling techniques used for PFAS site characterization are consistent with conventional sampling techniques used in the environmental industry, but special consideration is made regarding PFAS-containing materials and cross-contamination potential. **Table 1a** provides a summary of materials that have been approved for site investigation; this list is expected to grow longer as industry experience increases. **Table 1b** provides a summary of field equipment and materials that have available testing information and/or industry knowledge regarding

PFAS cross-contamination potential, and it is recommended that these materials be prohibited for sample collection; for materials that are suspected of containing PFAS and/or to retain PFAS, these recommendations are considered preliminary and subject to change. Further discussion of approved and prohibited materials is found throughout this document. In general, prohibited materials include but are not limited to peolytetrafluoroethylene (PFTE), waterproof coatings containing PFAS, fluorinated ethylene-propylene (FEP), ethylene tetrafluoroethylene (ETFE), low density polyethylene (LDPE), polyvinylidene fluoride (PVDF) [ITRC 2022].

Table 1a: Summary of Acceptable Sampling Equipment and Materials for PFAS Site Investigations

Sampling Materials	Additional Considerations	References
Water Sampling Materials		
High density polyethylene (HDPE) or silicone tubing materials	--	DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
HDPE HydraSleeves™	LDPE HydraSleeves™ are not recommended	USACE 2016; MassDEP 2017
Drilling and Soil Sampling Materials		
PFAS-free drilling fluids	--	DER 2016
PFAS-free makeup water	Confirm PFAS-free water source via laboratory analysis prior to investigation	--
Acetate liners	For use in soil sampling	USACE 2016
Sample Containers and Storage		
HDPE sample containers with HDPE lined lids for soil and water samples	Laboratory should provide; whole bottle analysis of aqueous samples combined with a solvent rinse of bottle is recommended	DER 2016, MassDEP 2017
Ice contained in plastic (polyethylene) bags (double bagged)	--	DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Field Documentation		
Ball point pens	--	MassDEP 2017
Standard paper and paper labels	--	DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Fine/Ultra-Fine point Sharpies®	Larger point Sharpies® should be avoided.	MDEQ 2018
Decontamination		

Sampling Materials	Additional Considerations	References
Water-only decontamination	Confirm PFAS-free water source via laboratory analysis prior to investigation	DER 2016
Alconox® or Liquinox® followed by deionized water or PFAS-free water rinse	Liquinox® known to contain trace levels of 1,4-dioxane	NHDES 2016; USACE 2016; MassDEP 2017
Sun and Biological Protection		
OFF Deep Woods, Sawyer Permethrin, Insect Shield® Permethrin	Apply >10 m away from sampling area	MDEQ 2018
Banana Boat, Coppertone, Neutrogena, Meijer, and L'Oreal Sunscreens	Apply >10 m away from sampling area	MDEQ 2018

Note: This list is considered preliminary and additional materials may be added as additional information becomes available. Project teams are expected to follow a methodical evaluation process of materials to be used and confirm acceptance prior to implementation of field activities.

Table 1b: Summary of Sampling Equipment and Materials Not Recommended for PFAS Site Investigations.

Sampling Materials	Known PFAS-Containing Materials	Suspected PFAS-Containing Materials	Materials with Potential to Retain PFAS	References
Water Sampling Materials				
Teflon®, PTFE-containing or other fluoropolymer coated or containing field equipment (e.g., tubing, bailers, liners, tape, plumbing paste, pump parts)	x			DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Passive diffusion bags			x	MassDEP 2017
LDPE HydraSleeves ™			x	USACE 2016; MassDEP 2017
Water particle filters			x	MassDEP 2017
Drilling and Soil Sampling Materials				
Aluminium foil			x	DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Drilling fluid containing PFAS	x	x		DER 2016
Coated bentonite		x		Radford et al 2023
Pipe thread compounds and tape			x	ITRC 2022

Sampling Materials	Known PFAS-Containing Materials	Suspected PFAS-Containing Materials	Materials with Potential to Retain PFAS	References
Sample Containers and Storage				
Glass sample containers with lined lids			x	DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
LDPE containers and lined lids			x	USACE 2016
Teflon® or PTFE- lined lids on containers (e.g., sample containers, rinsate water storage containers)	x			DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Reusable chemical or gel ice packs (e.g., BlueIce®)		x		DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Field Documentation				
Self-sticking notes and similar office products (e.g., 3M Post-it-notes)		x		DER 2016; USACE 2016; NHDES 2016; MassDEP 2017
Waterproof paper, notebooks, and labels	x			DER 2016, MassDEP 2017
Markers		x		NHDES 2016
Decontamination				
[Some] detergents and decontamination solutions (e.g., Decon 90® Decontamination Solution)	x	x		DER 2016; NHDES 2016; MassDEP 2017

Note: For materials that are suspected of containing PFAS, or have the potential to retain PFAS, project specific considerations may provide adequate justification for use during the field event. For example, further evaluation may be conducted in the form of pre-field equipment blank sample analysis.

Given the extremely low detection limits associated with PFAS analysis and the many potential sources of trace levels of PFAS, field personnel are advised to err on the side of caution by strictly following these protocols, frequently replacing nitrile gloves, and rinsing field equipment to help mitigate the potential for false detections of PFAS. A summary of other specific items related to field sampling for PFAS are discussed in the sections below.

This TGI applies to all Arcadis and subcontractor personnel involved in field sampling for PFAS.

4 Personnel Qualifications

4.1 Sampling Personnel

Field personnel must have current health and safety training, including 40-hour HAZWOPER training, up to date 8-hour refresher, site supervisor training, and site-specific training, as needed. In addition, field personnel will be versed in the other relevant SOPs (e.g., low flow sampling) and will possess the skills and experience necessary to successfully complete the desired field work. The site Health and Safety Plan (HASP) and other documents will identify any other training requirements such as site-specific safety training or access control requirements.

4.2 Laboratories

These laboratories are example laboratories that could be used to analyze environmental media for PFAS, pending project approval:

- United States: Pace, SGS, Vista, ALS, and Eurofins
- Canada: AXYS-SGS and Bureau Veritas

Other laboratories may be used if they are appropriately accredited for PFAS analysis according to any project requirements. It is recommended that a laboratory is Environmental Laboratory Accreditation Program (ELAP)-accredited for PFAS analysis in accordance with the Department of Defense (DoD) Quality Systems Manual (QSM) 5.3 Table B-15 or any subsequent updates. **For all data collection efforts at DoD sites, PFAS data must be obtained using a method that is DoD ELAP-accredited under QSM 5.3 or later.**

5 Equipment List

The following equipment and materials must be available for sampling:

- Site plan of sampling locations, relevant work plan (or equivalent), and this TGI;
- Appropriate health and safety equipment, as specified in the site HASP;
- Dedicated plastic sheeting (preferably high-density polyethylene [HDPE]) or other clean surface to prevent sample contact with the ground;
- Conductivity/temperature/pH meter;
- Dissolved oxygen meter, oxidation reduction potential meter, and turbidity meter;
- Depth to water meter;
- If using low-flow groundwater sampling techniques, peristaltic pump (groundwater sampling)/bladder pump (with PFAS free bladder/ HDPE bladder), flow through cell, and accompanying HDPE and silicone tubing;
- Hydrasleeves™, if using Hydrasleeves™ for groundwater sampling;
- Metal trowel for soil samples; specialized soil/sediment sampling equipment as required;
- Brushes for scrubbing sampling equipment;

- Pens, pencils, and/or fine/ultra-fine point Sharpies® for writing;
- Clipboards, field binders, and field note pages that are not waterproof;
- Labeled sample bottles:
    - There are various laboratory and analytical methods in place at the time of this TGI revision (Revision 11). Only use certified PFAS free materials provided by the laboratory for sample collection.
- If high concentrations of PFAS related to class B firefighting foams are expected, bring additional small vials to conduct field-based shaker tests for foaming;
- Ziploc® bags to hold ice and samples;
- Bottles containing “PFAS-free” water used for reagent blanks;
- Labeled, thoroughly decontaminated coolers for samples with ice; Blue ice is not permitted;
- Deionized or distilled water for initial decontamination rinsing;
- “PFAS-free” water provided by the laboratory for final decontamination rinsing;
- Alconox or Liquinox®;
- Packing and shipping materials;
- Groundwater and/or Sampling Log; and
- Chain-of-Custody (COC) Forms.

6 Cautions

6.1 Food Packaging

Some food packaging may be treated with PFAS-containing chemicals to prevent permeation of oil and water in the food outside of the packaging. To avoid potential food packaging-related PFAS contact:

- Do not bring any food outside of the field vehicles onsite and eat snacks and meals offsite.
- Wash hands after eating.
- Remove any field garments or outer layers prior to eating. Do not put them back on until done eating and hands are washed.

6.2 Field Gear

6.2.1 Clothing

Many types of clothing are treated with PFAS for stain and water resistance, in particular outdoor performance wear. To avoid potential clothing-related PFAS contact:

- Do not wear any outdoor performance wear that is water or stain resistant, or appears to be. Err on the side of caution.
- Wear pre-laundered (multiple washings, i.e., 6+) clothing that is not stain resistant or waterproof (unless made from the materials listed in Section 5.3.1).
- Natural fabrics such as cotton are preferred. Synthetic fabrics may also be acceptable if there is no indication on the label that the fabric is water and stain resistant.
- Most importantly, avoid contacting your clothing with sampling equipment, bottles, and samples.

6.2.2 Personal Protective Equipment

Safety Footwear

Some safety footwear has been treated to provide a degree of waterproofing and increased durability and may represent a source of trace PFAS. If at all possible, safety footwear without waterproofing should be worn; footwear that provides adequate safety from physical hazards is required and takes precedence over potential PFAS concerns. To avoid any PFAS cross contamination to samples from footwear:

- Do not contact your footwear with equipment, bottles, or samples in any way.
- Do not allow gloves used for sampling to come in contact with safety footwear.

Nitrile Gloves

Wear disposable nitrile gloves at all times. Don a new pair of nitrile gloves **before** the following activities at each sample location:

- Decontamination of re-usable sampling equipment;
- Contact with sample bottles or “PFAS-free” water bottles;
- Insertion of anything into the sample ports (e.g., HDPE tubing); and
- Handling of any quality assurance/quality control (QA/QC) samples including field blanks and equipment blanks.

Don a new pair of nitrile gloves **after** the following activities:

- Handling of any non-dedicated sampling equipment;
- Contact with contaminated surfaces; or
- When judged necessary by field personnel.

6.3 Personal Hygiene

- Shower at night.
- Do not use personal care products after showering such as lotions, makeup, and perfumes, UNLESS medically necessary.

- Use sunscreen and insect repellent as necessary for health and safety, i.e., if sampling is to occur outdoors in direct sunlight and/or if insect hazards may be present. Specific products that are acceptable for PFAS sampling are listed in Table 1 and in Section 6.1. Apply sunscreen and insect repellant prior to initiating field sampling. If sunscreen and/or repellant need to be reapplied, ensure a safe distance away from the sampling locations and equipment (i.e., more than 10 meters (m) away). Wash hands after application and don new gloves following hand washing.

6.4 Visitors

Visitors to the site are asked to remain at least 10 meters from sampling areas.

7 Health and Safety Considerations

7.1 Biological and Environmental Hazard Controls

7.1.1 Sunscreens and Insect Repellents

When site conditions warrant, insect repellent and sunscreen should be applied. Some insect repellents and sunscreen have been approved for PFAS sampling by individual states. According to Michigan Department of Environmental Quality (MDEQ; now known as Michigan Department of Environment, Great Lakes, and Energy [EGLE]), the products below are allowable (MDEQ 2018). Note that California State Water Quality Control Board's PFAS sampling guidance refers to MDEQ/EGLE's allowable list of sunscreens and insect repellents (California State Water Quality Control Board 2020).

Insect Repellents

- OFF Deep Woods
- Sawyer Permethrin

Sunscreen

- Banana Boat Sport Performance Sunscreen Lotion Broad Spectrum SPF 30
- Meijer Sunscreen Lotion Broad Spectrum SPF 30
- Neutrogena Ultra-Sheer Dry-Touch Sunscreen Broad Spectrum SPF 30
- Banana Boat for Men Triple Defense Continuous Spray Sunscreen SPF 30
- Banana Boat Sport Performance Coolzone Broad Spectrum SPF 30
- Banana Boat Sport Performance Sunscreen Lotion Broad Spectrum SPF 30
- Banana Boat Sport Performance Sunscreen Stick SPF 50
- Coppertone Sunscreen Lotion Ultra Guard Broad Spectrum SPF 50
- Coppertone Sport High-Performance AccuSpray Sunscreen SPF 30
- Coppertone Sunscreen Stick Kids SPF 55
- L'Oréal Silky Sheer Face Lotion 50+
- Meijer Clear Zinc Sunscreen Lotion Broad Spectrum SPF 15, 30 and 50
- Meijer Wet Skin Kids Sunscreen Continuous Spray Broad Spectrum SPF 70
- Neutrogena Beach Defense Water + Sun Barrier Lotion SPF 70

- Neutrogena Beach Defense Water + Sun Barrier Spray Broad Spectrum SPF 30
- Neutrogena Pure & Free Baby Sunscreen Broad Spectrum SPF 60+

Please plan for sampling events and purchase these products ahead of time. For any sunscreens and bug sprays, including those listed above, always follow these instructions for application:

- Insect repellents and sunscreen should be applied away from the work area prior to initiating sampling.
- When re-applying, stay at least 10 m away from the sampling locations and equipment.
- Wash hands after application and don new nitrile gloves.

7.1.2 Rain Event

Special care should be taken when rain is falling at the project site:

- Field sampling during extreme rainfall should be avoided if possible. If sampling needs to take place during a rain event (or other extreme weather condition), ensure the rain gear or other safety clothing is appropriate. For example, rain gear made from the following materials is allowable: polyurethane, PVC, wax coated fabrics, rubber/neoprene, uncoated Tyvek® (MDEQ 2018).
- If project timelines are tight, consider the use of a gazebo tent that can be erected over the top of the monitoring well to provide shelter from the rain. The canopy material is possibly a PFAS-treated surface and should be managed as such; therefore, wear gloves when moving the tent, change them immediately after moving the tent, and avoid further contact with the tent until all sampling activities have been finished and the team is ready to move on to the next site.

7.1.3 Other H&S Considerations

- ***If an unapproved or potentially suspect hazard control is needed for health and safety, apply or keep that control away from the samples, document its use in field notes, and, if it does contact a sample, take an equipment blank with that material.***
- The ability to safely access the surface water sampling locations must be verified before sampling.
- Field activities must be performed in accordance with the site HASP, a copy of which will be present onsite during such activities.
- Safety hazards associated with sampling surface water include fast-moving water, deep water, and steep slopes close to sampling sites. Use extreme caution when approaching sampling sites
- If thunder or lighting is present, discontinue sampling and take cover until 30 minutes have passed after the last occurrence of thunder or lighting.
- Use caution when removing well caps as well may be under pressure, cap can dislodge forcefully and cause injury.
- Avoid the use of anti-fog sprays on glasses, which may contain PFAS. It's recommended to instead purchase pre-treated anti-fog safety glasses.

8 Procedure

8.1 Field Equipment Cleaning

Reusable field sampling equipment will require cleaning before initial use and between uses. For groundwater sampling, between uses, decontaminate the flow-through cell and any non-dedicated equipment (i.e., interface probe of depth to water meter) that comes into contact with well water. Trowels and other materials used to sample soil samples will also require decontamination, although dedicated, single use equipment such as liners should be used where possible.

After donning a new pair of nitrile gloves:

- Rinse sampling equipment with Alconox or Liquinox® cleaning solution; Scrub equipment with a plastic brush if needed;
- Rinse with distilled water or deionized water;
- Rinse with “PFAS-free” water
- Repeat this entire process, scrubbing equipment a second time, rinsing with distilled or deionized water, then rinsing again with “PFAS-free” water (i.e., two scrubbings and four water rinsings total).
- Collect all rinsate in a sealed pail for disposal. Do not reuse decontamination solutions between sampling locations.

8.2 Borehole/Monitoring Well Development

If a drill rig is being used to drill for soil cores or to install monitoring wells, wear clean nitrile gloves before collecting each continuous soil sample. Additional requirements include the following:

- Verify in writing with the manufacturer that single-use liners used to collect each sample are made of a material that does not contain PFAS;
- Collect soil samples in laboratory-supplied HDPE bottles.
- Store the sample bottles in coolers and keep at a temperature of 0 to 6ºC until transported to the laboratory.

8.2.1 Well Condition Survey/ Water Level Monitoring

Using equipment that has been thoroughly decontaminated according to the procedures in Section 7.1, conduct the well condition surveys and water level monitoring:

- Conduct monitoring well inspections and record water levels.
- Use an interface probe to evaluate presence/absence of non-aqueous phase liquid (NAPL).
- Measure the depth to water from the top of the polyvinyl chloride (PVC) riser and the total depth of the well.
- Record information in the field notes.

8.2.2 Monitoring Well Development and Purging

Follow these requirements for monitoring well development and purging:

- Do not use Teflon™ tubing for purging or sample collection. HDPE tubing is acceptable.
- Do not re-use materials between wells. Upon completion of use, remove all disposable materials (such as HDPE and/or silicone tubing) and place in heavy duty garbage bags for disposal.
- During development of the well, create sufficient energy to agitate the water column and create flow reversals in the well screen, filter pack and formation to loosen fine-grained materials and draw them into the well. The pumping or bailing action should then draw all drilling fluids and fine-grained material out of the borehole and adjacent formation and then out of the well. Review the Arcadis Monitoring Well Development guidance (Arcadis 2010) for more detailed information.
- Follow the low-flow purge and sampling techniques per the U.S. Protection Agency's (EPA's) guidance document titled *Low Stress (Low Flow) purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells* (2010) and ASTM's standard titled *Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations* (2002). Also available for review is the Arcadis Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells (Arcadis 2011).
- To purge the well, if using HDPE tubing and a peristaltic pump, insert the end of the tubing to the approximate depth of the midpoint of the screened section of the monitoring wells. Measure the length of HDPE tubing to be inserted into each monitoring well and pre-cut it to approximate lengths (such as the previously measured arm span of a field technician) to avoid contact with any materials other than the monitoring well and peristaltic pump. Flow rates should be as low as can be reasonably achieved. Collect and appropriately dispose of purge water.
- Silicone tubing should direct the purge water through a flow-through cell for field parameter measurements of pH, conductivity, temperature, dissolved oxygen, and turbidity. Calibrate the instrument in the field prior to use.
- Record field parameters in intervals (generally of 3-minute duration) to ensure purge water has cycled through the flow-through cell. Sample the wells after field parameter measurements indicate stabilization, which allows collection of representative formation water (generally acceptable standards are three consecutive pH readings to within ±0.1 units, and three consecutive conductivity, temperature and dissolved oxygen measurements to within 3%). Turbidity must be monitored, but does not need to be used as a stabilization indicator of purge completion. Record field parameter measurements at each well. Drawdown should be monitored throughout the purge. Prior to collecting sample, disconnect tubing from flow-through cell and collect sample directly from tubing coming out of the monitoring well.
- If wells are suspected to be dewatering throughout the purge (i.e., reduced flow rate/difficulty pumping water or bubbles begin to come through the flow through cell), turn off the pump and allow the water level to recover for ½ hour, followed by sample collection. Document these activities in the field notes.

8.3 Sample Collection

Different laboratories may supply sample collection bottles of varying sizes depending on the type of media to be sampled. This section focuses on the collection of only soil, groundwater, surface water, and sediment. Different media may not follow these procedures. For example, AFFF concentrate should be sampled in accordance with DoD AFFF01 (DoD 2021).

8.3.1 Sample Containers

- Collect samples in HDPE bottles fitted with a HDPE lined (no Teflon™) screw cap.
- Complete bottle labels after the caps have been placed back on each bottle.
- Do not use glass bottles due to potential loss of analyte through adsorption. This is particularly important for aqueous samples.
- Review with analytical lab the sample size, sample container, etc. depending upon the type of PFAS analysis that is being requested.

8.3.2 Soil Sampling

Before Sample Collection

- Place LDPE plastic sheeting adjacent to the sample location for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.
- Trowels or drilling equipment that will come into contact with a sample should be decontaminated prior to sample collection, preferably with methanol/isopropanol/acetone;
- Don a new set of nitrile gloves. Do not use gloved hands to subsequently handle papers, pens, clothes, etc., before collecting samples.
- Use the HDPE bottles that are supplied by the laboratory. Make sure that the caps remain on the bottle until immediately prior to sample collection.

During Sample Collection

- Collect soil samples using a clean stainless-steel trowel or with single-use PFAS-free liners;
- Place soil samples in HDPE bottles supplied by the laboratory.
- Note the time on the sample label.
- Collect any necessary duplicates/co-located samples and matrix spikes – verify with laboratory whether they need to be collected in separate sample bottles.
- Collect any necessary equipment blanks. The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFAS, after the sampling equipment has been appropriately decontaminated.

- Collect any necessary field reagent blanks. This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel.

After Sample Collection

- Place each sample bottle in two sealed Ziploc® bags. Another brand of LDPE bag is acceptable.
- Record the label information and time of sampling in the field notes.
- Place soil sample bottles in coolers that are durable in transportation and keep the temperature between 0 and 6ºC until transported to the laboratory. Do not use blue ice.

8.3.3 Groundwater Sampling

Before Sample Collection

- Place LDPE plastic sheeting adjacent to the sample location for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.
- Don a new set of nitrile gloves. Do not use gloved hands to subsequently handle papers, pens, clothes, etc., before collecting samples.
- Use the HDPEbottles that are supplied by the laboratory. Make sure that the caps remain on the bottle until immediately prior to sample collection.
- Measure depth to water and field parameters. Turbidity and the physical appearance of the purged water should be noted on the Groundwater Sampling Log.

During Sample Collection

- Start groundwater sample collection upon stabilization of field parameters.
- If low-flow groundwater sampling techniques are being used, disconnect the silicone tubing from the flow-through cell, enabling collection of groundwater samples without passing through the cell.
- Hydrasleeves are also considered acceptable for sampling of PFAS in groundwater – consult the project manager to determine which technique should be used. In general, low flow sampling is preferable.
- Collect groundwater samples (to the neck of the bottle, some headspace is acceptable) from the dedicated sampling ports at the center of the well screen. While collecting the sample, make sure the bottle cap remains in the other hand of the sampler, until replaced on the bottle.
- To mitigate cross contamination, collect groundwater samples in a pre-determined order from least impacted to greater impacted based on previous analytical data or knowledge about past activities at the site. If no analytical data are available, samples are to be collected in the following order:
    1. First sample the upgradient well(s).
    2. Next, sample the well located furthest downgradient of the interpreted or known source.
    3. The remaining wells should be progressively sampled in order from downgradient to upgradient, such that the wells closest to the interpreted or known source are sampled last.

- NOTE: If high concentrations of PFAS related to class B firefighting foams are expected in a groundwater sample, conduct a Shaker test by collecting and shaking a small portion of the sample (~10 to 25 mL) on site in a small disposable vial. If foaming is noted within the sample, document the foaming when samples are submitted for analysis; the 'shaker test' vial can then be disposed. This shaker test provides information about how each of the samples should be handled analytically.
- After collecting the sample, tightly screw on the polypropylene cap (snug, but not too tight). This will minimize leaking or cross contamination of the sample.
- Note the time on the sample label.
- Collect any necessary duplicates and matrix spikes. As the laboratory should be analyzing the entire aqueous sample rather than sub-sampling, separate bottles will be required for these samples.
- Collect any necessary equipment blanks. The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFAS, after the sampling equipment has been appropriately decontaminated.
- Collect any necessary field reagent blanks. This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel.
- Do not rinse PFAS sample bottles during sampling. Do not filter samples.

After Sample Collection

- Place each sample bottle in two sealed Ziploc® bags. Another brand of LDPE bag is acceptable.
- Record the label information and time of sampling in the field notes and COC. Note ‘shake test’ results if appropriate.
- Place groundwater samples in coolers that are durable in transportation and keep the temperature between 0 and 6ºC until transported to the laboratory. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**

  Treat all disposable sampling materials as single use and dispose of them appropriately after sampling at each monitoring well.

8.3.4 Surface Water Sampling

Before Sample Collection

- Place LDPE plastic sheeting adjacent to the sample location for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.
- Don a new set of nitrile gloves. Do not use gloved hands to subsequently handle papers, pens, clothes, etc., before collecting samples.
- Use the HDPE bottles that are supplied by the laboratory. Make sure that the caps remain on the bottle until immediately prior to sample collection.

During Sample Collection

- Avoid sampling the surface, in general.
- However, for Air Force investigations, collect samples from the water surface.
- Where surface water samples and sediment samples are collected at the same location, collect surface water samples first to minimize siltation.
- Collect surface water samples directly into laboratory-supplied bottles; wide-mouth bottles may be preferable to narrow mouth bottles for ease of surface water collection.
- Make sure bottle caps remain in the gloved hand of the sampler until sampling is complete and caps are replaced on the bottle.
- Note the time on the sample bottle.
- Collect any necessary duplicates and matrix spikes. As the laboratory should be analyzing the entire aqueous sample rather than sub-sampling, separate bottles will be required for these samples.
- Collect any necessary equipment blanks. The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFAS, after the sampling equipment has been appropriately decontaminated.
- Collect any necessary field reagent blanks. This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel.

After Sample Collection

- Place each sample bottle in two sealed Ziploc® bags. Another brand of LDPE bag is acceptable.
- Record the label information and time of sampling in the field notes.
- Place samples in coolers that are durable in transportation and keep the temperature between 0 and 6ºC until transported to the laboratory. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**
- Measure surface water pH, conductivity, temperature, and TDS at each location **after** both surface water and sediment sampling.

8.3.5 Sediment Sampling

Before Sample Collection

- Place LDPE plastic sheeting adjacent to the sample location for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.
- Don a new set of nitrile gloves. Do not use gloved hands to subsequently handle papers, pens, clothes, etc., before collecting samples.
- Use the HDPE bottles that are supplied by the laboratory. Make sure that the caps remain on the bottle until immediately prior to sample collection.

During Sample Collection

- Where surface water samples and sediment samples are collected at the same location, collect surface water samples first to minimize siltation.
- Collect sediment samples either manually using a stainless-steel trowel or using a petite ponar grab sampler, depending on field conditions at each sampling location during sampling program.
- Collect sediment samples from the upper 10 cm of sediment.
- For a sample to be acceptable overlying, low turbidity water must be present.
- Decant the overlying water and use a stainless-steel trowel to collect only the upper 5 centimeters (cm) of sediment.
- Collect sediment samples directly into laboratory-supplied bottles that are suitable in both material and size.
- Do not overfill the sample bottle.
- Make sure that the sample does not contain vegetation, that the sediment is undisturbed, and that the sampler shows no signs of winnowing or leaking.
- Make sure bottle caps remain in the gloved hand of the sampler until sampling is complete and caps are replaced on the bottle.
- Note the time on the sample label.
- Collect any necessary duplicates and matrix spikes.
- Collect any necessary equipment blanks. The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFAS, after the sampling equipment has been appropriately decontaminated.
- Collect any necessary field reagent blanks. This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel.

After Sample Collection

- Place each sample bottle in two sealed Ziploc® bags. Another brand of LDPE bag is acceptable.
- Record the label information and time of sampling in the field notes.
- Place samples in coolers that are durable in transportation and keep the temperature between 0 and 6ºC until transported to the laboratory. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**
- Measure surface water pH, conductivity, temperature, and total dissolved solids (TDS) at each location **after** both surface water and sediment sampling is completed.

8.4 Shipping

- If samples cannot be shipped the same day as collected, arrange an appropriate means of keeping the samples cool overnight and maintain the temperature between 0 and 10°C for the first 48 hours after collection, and then between 0 and 6°C thereafter.

- Store samples in appropriate transport bottles (coolers) with ice (Ziploc© bags for use as ice containers) with appropriate labeling. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**
- Complete the appropriate procedures for COC, handling, packing, and shipping.
- Fill out and check COC Forms against the labels on the sample bottles progressively after each sample is collected.
- Place all disposable sampling materials (such as plastic sheeting, and health and safety equipment) in appropriate containers.
- Ship samples via courier service with priority overnight delivery. Tracking numbers for all shipments should be provided and recorded after they have been sent out to ensure their timely delivery.
- Confirm with the laboratory before shipping samples via Fed Ex for Saturday delivery.

9 Waste Management

All rinsate should be collected in a sealed pail for disposal. Drill cuttings and purge water will be managed as specified in the Field Sampling Plan (FSP) or Work Plan, and according to state and/or federal requirements. PPE and decontaminated fluids will be contained separately and staged at the sampling location. Containers must be labeled at the time of collection. Labels will include date, location(s), site name, city, state, and description of matrix contained (e.g., soil, groundwater, PPE). General guidelines for investigation derived waste (IDW) handling and storage are set forth in a separate IDW guidance document (Arcadis 2009).

Typical waste characterization procedures include collection of a composite sample of the drill cutting material and a composite sample of the purge water for laboratory analysis. Samples are typically analyzed for disposal toxicity characteristic leaching procedure (TCLP) analysis for metals and VOCs. For PFAS, a simple leach test with neutral pH water may be more indicative of actual risk. Additionally, generators of waste are required to include analysis of other constituents that are reasonably believed to be present including (in this case) PFAS.

Waste storage and final waste disposition should be determined in the site specific workplan.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

If digital data collection isn't possible, waterproof field books should be avoided for field notes. Instead, field notes on loose paper on Masonite, plastic, or aluminum clip boards is preferred. Please note that newer Rite in the Rain® notebooks are approved for PFAS sampling. Other requirements for field notes include:

- Pens, pencils, and fine/ultra-fine point Sharpies® may be used.
- Keep field notes and writing implements away from samples and sampling materials.
- One person should conduct sampling while another records field notes.
- Do not write on sampling bottles unless they are closed.

10.1 Other Project Documentation

- Complete groundwater and/or soil sampling logs.
- Make sure COC Forms are properly completed. Verify which PFAS analytes and methods (e.g., just PFOS/PFOA, some or all of the /1633 lists, etc.) are required for analysis and note on the COC.

11 Quality Assurance

Refer to quality control requirements for the project to ensure that appropriate quality assurance and quality control (QA/QC) samples are collected. When collecting QA/QC samples, the same guidelines apply as when collecting regular samples – specifically that:

- Samples should be collected in laboratory-supplied HDPE bottles;
- Bottle caps must remain in the hand of the sampler until replaced on the bottle;
- Labels must be completed after the caps have been placed back on each bottle; and
- Samples must be stored in appropriate transport bottles (coolers) with ice (Ziploc© bags for use as ice containers) with appropriate labeling. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**

11.1 Equipment Blanks (if relevant)

QA/QC sampling typically includes daily collection of equipment blanks using the laboratory-supplied “PFAS-free” water. For peristaltic pump tubing, laboratory supplied “PFAS-free” water should be poured into a clean HDPE sample bottle and then pumped through new HDPE tubing using the peristaltic pump (with new silicone tubing). The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFAS, after the sampling equipment has been appropriately decontaminated. Note that an equipment blank can also be collected if an unapproved or potentially suspect hazard control is needed for health and safety and it contacts a sample, i.e., that material would be exposed to PFAS free water then the water would be collected in a separate sample container.

11.2 Field Duplicates

QA/QC sampling typically includes the collection of one field duplicate for every 10 or 20 samples collected. Each duplicate sample will be collected immediately after the initial sample of which it is a duplicate into a separate laboratory-provided sample bottle. Do not indicate to the laboratory which sample the duplicate replicates, i.e., it should be given a blind reference on the COC and sample name such as “Duplicate-01”.

11.3 Field Reagent Blanks

QA/QC sampling for PFAS typically includes the submission of one laboratory supplied field reagent blank per day. The field reagent blank sample is brought to the site in a laboratory-supplied sample bottle. Field staff transfer the laboratory-supplied reagent blank to an empty sample bottle. This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel and should be placed in the same cooler as the other PFAS samples.

11.4 Matrix Spikes (optional in some cases)

QA/QC sampling includes submitting samples to be used as a matrix spike and matrix spike duplicate (MS/MSD) if the project requires it. If separate sample bottles are required, additional samples will be collected immediately after the initial sample of which it is a duplicate into separate laboratory-supplied sample bottles.

11.5 Laboratory Analytical QA/QC

- Arcadis recommends that any request for PFAS analysis in groundwater or soil should be conducted by an ELAP-accredited method compliant with QSM 5.3 Table B-15. Requirements laid out in Table B-15 strictly govern acceptable laboratory data quality for PFAS analysis in environmental samples. **For all data collection efforts at DoD sites, PFAS data must be obtained using a method that is DoD ELAP-accredited under QSM 5.3 or later.**
- Laboratory QA/QC should consist of one laboratory blank and one laboratory control sample (or blank spike) per batch of samples, and additional QA/QCs as indicated by the laboratory QA/QC procedures.
- Isotope dilution must be used for quantification with isotope-labeled spiked standards (extracted internal standards EIS), as available, according to the guidelines of QSM 5.3 Table B-15. See EPA method 1633 and DoD QSM 5.4 Table B-24 for quantification requirements by isotope dilution with EIS standards. The USEPA has two drinking water methods (USEPA Method 537.1 and USEPA Method 533).
- For drinking water, groundwater, and surface water samples, laboratories must extract the entire sample and include a solvent rinse of the bottle for analysis. Aqueous samples should generally not be sub-sampled prior to analysis, unless they are high concentration and require serial dilution (US DoD 2017).
- Soil samples should be analyzed in their entirety or thoroughly homogenized before extraction and analysis.
- As part of the internal QA/QC of laboratory results, relative percent difference (RPD) should be calculated between samples and corresponding field or laboratory duplicates. The laboratory quality assurance portion of the laboratory certificates should be reviewed to verify that all calculations/recoveries were within acceptable limits as established by the laboratory method and guidelines in Table B-15 of QSM 5.3 or later (USDoD 2019).

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Department of Environmental Conservation

SAMPLING, ANALYSIS, AND ASSESSMENT OF PER- AND POLYFLUOROALKYL SUBSTANCES (PFAS)

Under NYSDEC's Part 375 Remedial Programs

April 2023



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ERRATA SHEET for

SAMPLING, ANALYSIS, AND ASSESSMENT OF PER- AND POLYFLUOROALKYL SUBSTANCES (PFAS) Under NYSDEC's Part 375 Remedial Programs Issued January 17, 2020

Citation and Page Number	Current Text	Corrected Text	Date
Title of Appendix I, page 32	Appendix H	Appendix I	2/25/2020
Document Cover, page 1	Guidelines for Sampling and Analysis of PFAS	Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) Under NYSDEC's Part 375 Remedial Programs	9/15/2020
Data Assessment and Application to Site Cleanup Page 3	Until such time as Ambient Water Quality Standards (AWQS) and Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published	Until such time as Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published	3/28/2023
Water Sample Results Page 3	PFOA and PFOS should be further assessed and considered as potential contaminants of concern in groundwater or surface water if PFOA or PFOS is detected in any water sample at or above 10 ng/L (ppt) and is determined to be attributable to the site, either by a comparison of upgradient and downgradient levels, or the presence of soil source areas, as defined below.	NYSDEC has adopted ambient water quality guidance values for PFOA and PFOS. Groundwater samples should be compared to the human health criteria of 6.7 ng/l (ppt) for PFOA and 2.7 ng/l (ppt) for PFOS. These guidance values also include criteria for surface water for PFOS applicable for aquatic life, which may be applicable at some sites. Drinking water sample results should be compared to the NYS maximum contaminant level (MCL) of 10 ng/l (ppt).Analysis to determine if PFOA and PFOS concentrations are attributable to the site should include a comparison between upgradient and downgradient levels, and the presence of soil source areas, as defined below.	3/28/2023
Soil Sample Results Page 3	Soil cleanup objectives for PFOA and PFOS have been proposed in an upcoming revision to 6 NYCRR Part 375-6. Until SCOs are in effect, the following are to be used as guidance values:	NYSDEC will delay adding soil cleanup objectives for PFOA and PFOS to 6 NYCRR Part 375-6 until the PFAS rural soil background study has been completed. Until SCOs are in effect, the following are to be used as guidance values:	3/28/2023
Protection of Groundwater Page 3	PFOA (ppb) 1.1 PFOS (ppb) 3.7	PFOA (ppb) 0.8 PFOS (ppb) 1.0	3/28/2023

Citation and Page Number	Current Text	Corrected Text	Date
Footnote 2 Page 3	The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the guidance value for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf).	The Protection of Groundwater values are based on the above referenced ambient groundwater guidance values. Details on that calculation are available in the following document, prepared for the February 2022 proposed changes to Part 375 (https://www.dec.ny.gov/docs/remediation_hudson_pdf/part375techsupport.pdf). The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the guidance value for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf).	3/28/2023
Testing for Imported Soil Page 4	If the concentrations of PFOA and PFOS in leachate are at or above 10 ppt (the Maximum Contaminant Levels established for drinking water by the New York State Department of Health), then the soil is not acceptable.	If the concentrations of PFOA and PFOS in leachate are at or above the ambient water quality guidance values for groundwater, then the soil is not acceptable.	3/28/2023
Routine Analysis, page 9	"However, laboratories analyzing environmental samples…PFOA and PFOS in drinking water by EPA Method 537, 537.1 or ISO 25101."	"However, laboratories analyzing environmental samples…PFOA and PFOS in drinking water by EPA Method 537, 537.1, ISO 25101, or Method 533."	9/15/2020
Additional Analysis, page 9, new paragraph regarding soil parameters	None	"In cases where site-specific cleanup objectives for PFOA and PFOS are to be assessed, soil parameters, such as Total Organic Carbon (EPA Method 9060), soil pH (EPA Method 9045), clay content (percent), and cation exchange capacity (EPA Method 9081), should be included in the analysis to help evaluate factors affecting the leachability of PFAS in site soils."	9/15/2020

Citation and Page Number	Current Text	Corrected Text	Date
Data Assessment and Application to Site Cleanup Page 10	Until such time as Ambient Water Quality Standards (AWQS) and Soil Cleanup Objectives (SCOs) for PFAS are published, the extent of contaminated media potentially subject to remediation should be determined on a case-by-case basis using the procedures discussed below and the criteria in DER-10. Target levels for cleanup of PFAS in other media, including biota and sediment, have not yet been established by the DEC.	Until such time as Ambient Water Quality Standards (AWQS) and Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published, the extent of contaminated media potentially subject to remediation should be determined on a case-by-case basis using the procedures discussed below and the criteria in DER-10. Preliminary target levels for cleanup of PFOA and PFOS in other media, including biota and sediment, have not yet been established by the DEC.	9/15/2020
Water Sample Results Page 10	PFAS should be further assessed and considered as a potential contaminant of concern in groundwater or surface water (…) If PFAS are identified as a contaminant of concern for a site, they should be assessed as part of the remedy selection process in accordance with Part 375 and DER-10.	PFOA and PFOS should be further assessed and considered as potential contaminants of concern in groundwater or surface water (…) If PFOA and/or PFOS are identified as contaminants of concern for a site, they should be assessed as part of the remedy selection process in accordance with Part 375 and DER-10.	9/15/2020

Citation and Page Number	Current Text	Corrected Text	Date
Soil Sample Results, page 10	"The extent of soil contamination for purposes of delineation and remedy selection should be determined by having certain soil samples tested by Synthetic Precipitation Leaching Procedure (SPLP) and the leachate analyzed for PFAS. Soil exhibiting SPLP results above 70 ppt for either PFOA or PFOS (individually or combined) are to be evaluated during the cleanup phase."	"Soil cleanup objectives for PFOA and PFOS will be proposed in an upcoming revision to 6 NYCRR Part 375-6. Until SCOs are in effect, the following are to be used as guidance values. " [Interim SCO Table] "PFOA and PFOS results for soil are to be compared against the guidance values listed above. These guidance values are to be used in determining whether PFOA and PFOS are contaminants of concern for the site and for determining remedial action objectives and cleanup requirements. Site-specific remedial objectives for protection of groundwater can also be presented for evaluation by DEC. Development of site-specific remedial objectives for protection of groundwater will require analysis of additional soil parameters relating to leachability. These additional analyses can include any or all the parameters listed above (soil pH, cation exchange capacity, etc.) and/or use of SPLP. As the understanding of PFAS transport improves, DEC welcomes proposals for site-specific remedial objectives for protection of groundwater. DEC will expect that those may be dependent on additional factors including soil pH, aqueous pH, % organic carbon, % Sand/Silt/Clay, soil cations: K, Ca, Mg, Na, Fe, Al, cation exchange capacity, and anion exchange capacity. Site-specific remedial objectives should also consider the dilution attenuation factor (DAF). The NJDEP publication on DAF can be used as a reference: https://www.nj.gov/dep/srp/guidance/rs/daf.pdf. "	9/15/2020

Citation and Page Number	Current Text	Corrected Text	Date
Testing for Imported Soil Page 11	Soil imported to a site for use in a soil cap, soil cover, or as backfill is to be tested for PFAS in general conformance with DER-10, Section 5.4(e) for the PFAS Analyte List (Appendix F) using the analytical procedures discussed below and the criteria in DER-10 associated with SVOCs. If PFOA or PFOS is detected in any sample at or above 1 µg/kg, then soil should be tested by SPLP and the leachate analyzed for PFAS. If the SPLP results exceed 10 ppt for either PFOA or PFOS (individually) then the source of backfill should be rejected, unless a site-specific exemption is provided by DER. SPLP leachate criteria is based on the Maximum Contaminant Levels proposed for drinking water by New York State's Department of Health, this value may be updated based on future Federal or State promulgated regulatory standards. Remedial parties have the option of analyzing samples concurrently for both PFAS in soil and in the SPLP leachate to minimize project delays. Category B deliverables should be submitted for backfill samples, though a DUSR is not required.	Testing for PFAS should be included any time a full TAL/TCL analyte list is required. Results for PFOA and PFOS should be compared to the applicable guidance values. If PFOA or PFOS is detected in any sample at or above the guidance values then the source of backfill should be rejected, unless a site-specific exemption is provided by DER based on SPLP testing, for example. If the concentrations of PFOA and PFOS in leachate are at or above 10 ppt (the Maximum Contaminant Levels established for drinking water by the New York State Department of Health), then the soil is not acceptable. PFOA, PFOS and 1,4-dioxane are all considered semi-volatile compounds, so composite samples are appropriate for these compounds when sampling in accordance with DER-10, Table 5.4(e)10. Category B deliverables should be submitted for backfill samples, though a DUSR is not required.	9/15/2020

Citation and Page Number	Current Text	Corrected Text	Date
Footnotes	None	[1] TOP Assay analysis of highly contaminated samples, such as those from an AFFF (aqueous film-forming foam) site, can result in incomplete oxidation of the samples and an underestimation of the total perfluoroalkyl substances. [2] The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the soil cleanup objective for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf).	9/15/2020
Additional Analysis, page 9	In cases… soil parameters, such as Total Organic Carbon (EPA Method 9060), soil…	In cases… soil parameters, such as Total Organic Carbon (Lloyd Kahn), soil…	1/8/2021
Appendix A, General Guidelines, fourth bullet	List the ELAP-approved lab(s) to be used for analysis of samples	List the ELAP- certified lab(s) to be used for analysis of samples	1/8/2021
Appendix E, Laboratory Analysis and Containers	Drinking water samples collected using this protocol are intended to be analyzed for PFAS by ISO Method 25101.	Drinking water samples collected using this protocol are intended to be analyzed for PFAS by EPA Method 537, 537.1, 533, or ISO Method 25101	1/8/2021
Water Sample Results Page 9	"In addition, further assessment of water may be warranted if either of the following screening levels are met: a. any other individual PFAS (not PFOA or PFOS) is detected in water at or above 100 ng/L; or b. total concentration of PFAS (including PFOA and PFOS) is detected in water at or above 500 ng/L"	Deleted	6/15/2021

Citation and Page Number	Current Text	Corrected Text	Date
Routine Analysis, Page XX	Currently, New York State Department of Health's Environmental Laboratory Approval Program (ELAP)… criteria set forth in the DER's laboratory guidelines for PFAS in non-potable water and solids (Appendix H - Laboratory Guidelines for Analysis of PFAS in Non-Potable Water and Solids).	Deleted	5/31/2022
Analysis and Reporting, Page XX	As of October 2020, the United States Environmental Protection Agency (EPA) does not have a validated method for analysis of PFAS for media commonly analyzed under DER remedial programs (non-potable waters, solids). DER has developed the following guidelines to ensure consistency in analysis and reporting of PFAS.	Deleted	5/31/2022
Routine Analysis, Page XX	LC-MS/MS analysis for PFAS using methodologies based on EPA Method 537.1 is the procedure to use for environmental samples. Isotope dilution techniques should be utilized for the analysis of PFAS in all media.	EPA Method 1633 is the procedure to use for environmental samples.	
Soil Sample Results, Page XX	Soil cleanup objectives for PFOA and PFOS will be proposed in an upcoming revision to 6 NYCRR Part 375-6	Soil cleanup objectives for PFOA and PFOS have been proposed in an upcoming revision to 6 NYCRR Part 375-6	
Appendix A	"Include in the text… LC-MS/MS for PFAS using methodologies based on EPA Method 537.1"	"Include in the text ….EPA Method 1633"	
Appendix A	"Laboratory should have ELAP certification for PFOA and PFOS in drinking water by EPA Method 537, 537.1, EPA Method 533, or ISO 25101"	Deleted	
Appendix B	"Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1"	"Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633"	

Citation and Page Number	Current Text	Corrected Text	Date
Appendix C	"Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1"	"Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633"	
Appendix D	"Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1"	"Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633"	
Appendix G		Updated to include all forty PFAS analytes in EPA Method 533	
Appendix H		Deleted	
Appendix I	Appendix I	Appendix H	
Appendix H	"These guidelines are intended to be used for the validation of PFAS analytical results for projects within the Division of Environmental Remediation (DER) as well as aid in the preparation of a data usability summary report."	"These guidelines are intended to be used for the validation of PFAS using EPA Method 1633 for projects within the Division of Environmental Remediation (DER)."	
Appendix H	"The holding time is 14 days…"	"The holding time is 28 days…"	
Appendix H, Initial Calibration	"The initial calibration should contain a minimum of five standards for linear fit…"	"The initial calibration should contain a minimum of six standards for linear fit…"	
Appendix H, Initial Calibration	Linear fit calibration curves should have an R^2 value greater than 0.990.	Deleted	
Appendix H, Initial Calibration Verification	Initial Calibration Verification Section	Deleted	
Appendix H	secondary Ion Monitoring Section	Deleted	
Appendix H	Branched and Linear Isomers Section	Deleted	

Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) Under NYSDEC's Part 375 Remedial Programs

Objective

New York State Department of Environmental Conservation's Division of Environmental Remediation (DER) performs or oversees sampling of environmental media and subsequent analysis of PFAS as part of remedial programs implemented under 6 NYCRR Part 375. To ensure consistency in sampling, analysis, reporting, and assessment of PFAS, DER has developed this document which summarizes currently accepted procedures and updates previous DER technical guidance pertaining to PFAS.

Applicability

All work plans submitted to DEC pursuant to one of the remedial programs under Part 375 shall include PFAS sampling and analysis procedures that conform to the guidelines provided herein.

As part of a site investigation or remedial action compliance program, whenever samples of potentially affected media are collected and analyzed for the standard Target Analyte List/Target Compound List (TAL/TCL), PFAS analysis should also be performed. Potentially affected media can include soil, groundwater, surface water, and sediment. Based upon the potential for biota to be affected, biota sampling and analysis for PFAS may also be warranted as determined pursuant to a Fish and Wildlife Impact Analysis. Soil vapor sampling for PFAS is not required.

Field Sampling Procedures

DER-10 specifies technical guidance applicable to DER's remedial programs. Given the prevalence and use of PFAS, DER has developed "best management practices" specific to sampling for PFAS. As specified in DER-10 Chapter 2, quality assurance procedures are to be submitted with investigation work plans. Typically, these procedures are incorporated into a work plan, or submitted as a stand-alone document (e.g., a Quality Assurance Project Plan). Quality assurance guidelines for PFAS are listed in Appendix A - Quality Assurance Project Plan (QAPP) Guidelines for PFAS.

Field sampling for PFAS performed under DER remedial programs should follow the appropriate procedures outlined for soils, sediments, or other solids (Appendix B), non-potable groundwater (Appendix C), surface water (Appendix D), public or private water supply wells (Appendix E), and fish tissue (Appendix F).

QA/QC samples (e.g. duplicates, MS/MSD) should be collected as specified in DER-10, Section 2.3(c). For sampling equipment coming in contact with aqueous samples only, rinsate or equipment blanks should be collected. Equipment blanks should be collected at a minimum frequency of one per day per site or one per twenty samples, whichever is more frequent.

Analysis and Reporting

The investigation work plan should describe analysis and reporting procedures, including laboratory analytical procedures for the methods discussed below. As specified in DER-10 Section 2.2, laboratories should provide a full Category B deliverable. In addition, a Data Usability Summary Report (DUSR) should be prepared by an independent, third-party data validator. Electronic data submissions should meet the requirements provided at: https://www.dec.ny.gov/chemical/62440.html.

DER has developed a *PFAS Analyte List* (Appendix G) for remedial programs to understand the nature of contamination at sites. It is expected that reported results for PFAS will include, at a minimum, all the compounds listed. If lab and/or matrix specific issues are encountered for any analytes, the DER project manager, in consultation with the DER chemist, will make case-by-case decisions as to whether certain analytes may be temporarily or permanently discontinued from analysis at each site. As with other contaminants that are analyzed for at a site, the *PFAS Analyte List* may be refined for future sampling events based on investigative findings.

Routine Analysis

EPA Method 1633 is the procedure to use for environmental samples. Reporting limits for PFOA and PFOS in aqueous samples should not exceed 2 ng/L. Reporting limits for PFOA and PFOS in solid samples should not exceed 0.5 µg/kg. Reporting limits for all other PFAS in aqueous and solid media should be as close to these limits as possible. If laboratories indicate that they are not able to achieve these reporting limits for the entire *PFAS Analyte List*, site-specific decisions regarding acceptance of elevated reporting limits for specific PFAS can be made by the DER project manager in consultation with the DER chemist. Data review guidelines were developed by DER to ensure data comparability and usability (Appendix H - Data Review Guidelines for Analysis of PFAS in Non-Potable Water and Solids).

Additional Analysis

Additional laboratory methods for analysis of PFAS may be warranted at a site, such as the Synthetic Precipitation Leaching Procedure (SPLP) and Total Oxidizable Precursor Assay (TOP Assay).

In cases where site-specific cleanup objectives for PFOA and PFOS are to be assessed, soil parameters, such as Total Organic Carbon (Lloyd Kahn), soil pH (EPA Method 9045), clay content (percent), and cation exchange capacity (EPA Method 9081), should be included in the analysis to help evaluate factors affecting the leachability of PFAS in site soils.

SPLP is a technique used to determine the mobility of chemicals in liquids, soils and wastes, and may be useful in determining the need for addressing PFAS-containing material as part of the remedy. SPLP by EPA Method 1312 should be used unless otherwise specified by the DER project manager in consultation with the DER chemist.

Impacted materials can be made up of PFAS that are not analyzable by routine analytical methodology. A TOP Assay can be utilized to conceptualize the amount and type of oxidizable PFAS which could be liberated in the environment, which approximates the maximum concentration of perfluoroalkyl substances that could be generated if all polyfluoroalkyl substances were oxidized. For example, some polyfluoroalkyl substances may degrade or transform to form perfluoroalkyl substances (such as PFOA or PFOS), resulting in an increase in perfluoroalkyl substance concentrations as contaminated groundwater moves away from a source. The TOP Assay converts, through oxidation, polyfluoroalkyl substances (precursors) into perfluoroalkyl substances that can be detected by routine analytical methodology.[1]

[1] TOP Assay analysis of highly contaminated samples, such as those from an AFFF (aqueous film-forming foam) site, can result in incomplete oxidation of the samples and an underestimation of the total perfluoroalkyl substances.

Commercial laboratories have adopted methods which allow for the quantification of targeted PFAS in air and biota. The EPA's Office of Research and Development (ORD) is currently developing methods which allow for air emissions characterization of PFAS, including both targeted and non-targeted analysis of PFAS. Consult with the DER project manager and the DER chemist for assistance on analyzing biota/tissue and air samples.

Data Assessment and Application to Site Cleanup

Until such time as Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published, the extent of contaminated media potentially subject to remediation should be determined on a case-by-case basis using the procedures discussed below and the criteria in DER-10. Preliminary target levels for cleanup of PFOA and PFOS in other media, including biota and sediment, have not yet been established by the DEC.

Water Sample Results

NYSDEC has adopted ambient water quality guidance values for PFOA and PFOS. Groundwater samples should be compared to the human health criteria of 6.7 ng/l (ppt) for PFOA and 2.7 ng/l (ppt) for PFOS. These human health criteria should also be applied to surface water that is used as a water supply. This guidance also includes criteria for surface water for PFOS applicable for aquatic life, which may be applicable at some sites. Drinking water sample results should be compared to the NYS maximum contaminant level (MCL) of 10 ng/l (ppt). Analysis to determine if PFOA and PFOS concentrations are attributable to the site should include a comparison between upgradient and downgradient levels, and the presence of soil source areas, as defined below.

If PFOA and/or PFOS are identified as contaminants of concern for a site, they should be assessed as part of the remedy selection process in accordance with Part 375 and DER-10.

Soil Sample Results

NYSDEC will delay adding soil cleanup objectives for PFOA and PFOS to 6 NYCRR Part 375-6 until the PFAS rural soil background study has been completed. Until SCOs are in effect, the following are to be used as guidance values:

Guidance Values for Anticipated Site Use	PFOA (ppb)	PFOS (ppb)
Unrestricted	0.66	0.88
Residential	6.6	8.8
Restricted Residential	33	44
Commercial	500	440
Industrial	600	440
Protection of Groundwater[2]	0.8	1.0

PFOA and PFOS results for soil are to be compared against the guidance values listed above. These guidance values are to be used in determining whether PFOA and PFOS are contaminants of concern for the site and for determining remedial action objectives and cleanup requirements. Site-specific remedial objectives for protection of groundwater can also be presented for evaluation by DEC. Development of site-specific remedial objectives for protection of groundwater will require analysis of additional soil parameters relating to leachability. These

[2] The Protection of Groundwater values are based on the above referenced ambient groundwater guidance values. Details on that calculation are available in the following document, prepared for the February 2022 proposed changes to Part 375 (https://www.dec.ny.gov/docs/remediation_hudson_pdf/part375techsupport.pdf). The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the guidance value for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf).

additional analyses can include any or all the parameters listed above (soil pH, cation exchange capacity, etc.) and/or use of SPLP.

As the understanding of PFAS transport improves, DEC welcomes proposals for site-specific remedial objectives for protection of groundwater. DEC will expect that those may be dependent on additional factors including soil pH, aqueous pH, % organic carbon, % Sand/Silt/Clay, soil cations: K, Ca, Mg, Na, Fe, Al, cation exchange capacity, and anion exchange capacity. Site-specific remedial objectives should also consider the dilution attenuation factor (DAF). The NJDEP publication on DAF can be used as a reference: https://www.nj.gov/dep/srp/guidance/rs/daf.pdf.

Testing for Imported Soil

Testing for PFAS should be included any time a full TAL/TCL analyte list is required. Results for PFOA and PFOS should be compared to the applicable guidance values. If PFOA or PFOS is detected in any sample at or above the guidance values then the source of backfill should be rejected, unless a site-specific exemption is provided by DER based on SPLP testing, for example. If the concentrations of PFOA and PFOS in leachate are at or above the ambient water quality guidance values for groundwater, then the soil is not acceptable.

PFOA, PFOS and 1,4-dioxane are all considered semi-volatile compounds, so composite samples are appropriate for these compounds when sampling in accordance with DER-10, Table 5.4(e)10. Category B deliverables should be submitted for backfill samples, though a DUSR is not required.

Appendix A - Quality Assurance Project Plan (QAPP) Guidelines for PFAS

The following guidelines (general and PFAS-specific) can be used to assist with the development of a QAPP for projects within DER involving sampling and analysis of PFAS.

General Guidelines in Accordance with DER-10

- Document/work plan section title – Quality Assurance Project Plan
- Summarize project scope, goals, and objectives
- Provide project organization including names and resumes of the project manager, Quality Assurance Officer (QAO), field staff, and Data Validator
    - The QAO should not have another position on the project, such as project or task manager, that involves project productivity or profitability as a job performance criterion
- List the ELAP certified lab(s) to be used for analysis of samples
- Include a site map showing sample locations
- Provide detailed sampling procedures for each matrix
- Include Data Quality Usability Objectives
- List equipment decontamination procedures
- Include an "Analytical Methods/Quality Assurance Summary Table" specifying:
    - Matrix type
    - Number or frequency of samples to be collected per matrix
    - Number of field and trip blanks per matrix
    - Analytical parameters to be measured per matrix
    - Analytical methods to be used per matrix with minimum reporting limits
    - Number and type of matrix spike and matrix spike duplicate samples to be collected
    - Number and type of duplicate samples to be collected
    - Sample preservation to be used per analytical method and sample matrix
    - Sample container volume and type to be used per analytical method and sample matrix
    - Sample holding time to be used per analytical method and sample matrix
- Specify Category B laboratory data deliverables and preparation of a DUSR

Specific Guidelines for PFAS

- Include in the text that sampling for PFAS will take place
- Include in the text that PFAS will be analyzed by EPA Method 1633
- Include the list of PFAS compounds to be analyzed (*PFAS Analyte List*)
- Include the laboratory SOP for PFAS analysis
- List the minimum method-achievable Reporting Limits for PFAS
    - Reporting Limits should be less than or equal to:
        - Aqueous – 2 ng/L (ppt)
        - Solids – 0.5 µg/kg (ppb)
- Include the laboratory Method Detection Limits for the PFAS compounds to be analyzed
-
- Include detailed sampling procedures
    - Precautions to be taken
    - Pump and equipment types
    - Decontamination procedures
    - Approved materials only to be used
- Specify that regular ice only will be used for sample shipment
- Specify that equipment blanks should be collected at a minimum frequency of 1 per day per site for each matrix

Appendix B - Sampling Protocols for PFAS in Soils, Sediments and Solids

General

The objective of this protocol is to give general guidelines for the collection of soil, sediment and other solid samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Containers

Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633.

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment

Acceptable materials for sampling include stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in to contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel spoon
- stainless steel bowl
- steel hand auger or shovel without any coatings

Equipment Decontamination

Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

Sampling Techniques

Sampling is often conducted in areas where a vegetative turf has been established. In these cases, a pre-cleaned trowel or shovel should be used to carefully remove the turf so that it may be replaced at the conclusion of sampling. Surface soil samples (e.g. 0 to 6 inches below surface) should then be collected using a pre-cleaned, stainless steel spoon. Shallow subsurface soil samples (e.g. 6 to ~36 inches below surface) may be collected by digging a hole using a pre-cleaned hand auger or shovel. When the desired subsurface depth is reached, a pre-cleaned hand auger or spoon shall be used to obtain the sample.

When the sample is obtained, it should be deposited into a stainless steel bowl for mixing prior to filling the sample containers. The soil should be placed directly into the bowl and mixed thoroughly by rolling the material into the middle until the material is homogenized. At this point the material within the bowl can be placed into the laboratory provided container.

Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at 4 ± 2º Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- Request appropriate data deliverable (Category B) and an electronic data deliverable

Documentation

A soil log or sample log shall document the location of the sample/borehole, depth of the sample, sampling equipment, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.

Appendix C - Sampling Protocols for PFAS in Monitoring Wells

General

The objective of this protocol is to give general guidelines for the collection of groundwater samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Container

Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633.

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment

Acceptable materials for sampling include: stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including plumbers tape and sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel inertia pump with HDPE tubing
- peristaltic pump equipped with HDPE tubing and silicone tubing
- stainless steel bailer with stainless steel ball
- bladder pump (identified as PFAS-free) with HDPE tubing

Equipment Decontamination

Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

Sampling Techniques

Monitoring wells should be purged in accordance with the sampling procedure (standard/volume purge or low flow purge) identified in the site work plan, which will determine the appropriate time to collect the sample. If sampling using standard purge techniques, additional purging may be needed to reduce turbidity levels, so samples contain a limited amount of sediment within the sample containers. Sample containers that contain sediment may cause issues at the laboratory, which may result in elevated reporting limits and other issues during the sample preparation that can compromise data usability. Sampling personnel should don new nitrile gloves prior to sample collection due to the potential to contact PFAS containing items (not related to the sampling equipment) during the purging activities.

Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at 4 ± 2º Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- Collect one equipment blank per day per site and minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers
- Additional equipment blank samples may be collected to assess other equipment that is utilized at the monitoring well
- Request appropriate data deliverable (Category B) and an electronic data deliverable

Documentation

A purge log shall document the location of the sample, sampling equipment, groundwater parameters, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.

Appendix D - Sampling Protocols for PFAS in Surface Water

General

The objective of this protocol is to give general guidelines for the collection of surface water samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Container

Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633.

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment

Acceptable materials for sampling include: stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel cup

Equipment Decontamination

Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

Sampling Techniques

Where conditions permit, (e.g. creek or pond) sampling devices (e.g. stainless steel cup) should be rinsed with site medium to be sampled prior to collection of the sample. At this point the sample can be collected and poured into the sample container.

If site conditions permit, samples can be collected directly into the laboratory container.

Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at 4 ± 2º Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- Collect one equipment blank per day per site and minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers
- Request appropriate data deliverable (Category B) and an electronic data deliverable

Documentation

A sample log shall document the location of the sample, sampling equipment, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.

Appendix E - Sampling Protocols for PFAS in Private Water Supply Wells

General

The objective of this protocol is to give general guidelines for the collection of water samples from private water supply wells (with a functioning pump) for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

Laboratory Analysis and Container

Drinking water samples collected using this protocol are intended to be analyzed for PFAS by EPA Method 537, 537.1, 533, or ISO Method 25101. The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

Equipment

Acceptable materials for sampling include stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials (e.g. plumbers tape), including sample bottle cap liners with a PTFE layer.

Equipment Decontamination

Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

Sampling Techniques

Locate and assess the pressure tank and determine if any filter units are present within the building. Establish the sample location as close to the well pump as possible, which is typically the spigot at the pressure tank. Ensure sampling equipment is kept clean during sampling as access to the pressure tank spigot, which is likely located close to the ground, may be obstructed and may hinder sample collection.

Prior to sampling, a faucet downstream of the pressure tank (e.g., washroom sink) should be run until the well pump comes on and a decrease in water temperature is noted which indicates that the water is coming from the well. If the homeowner is amenable, staff should run the water longer to purge the well (15+ minutes) to provide a sample representative of the water in the formation rather than standing water in the well and piping system including the pressure tank. At this point a new pair of nitrile gloves should be donned and the sample can be collected from the sample point at the pressure tank.

Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at 4 ± 2º Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- If equipment was used, collect one equipment blank per day per site and a minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers.
- A field reagent blank (FRB) should be collected at a rate of one per 20 samples. The lab will provide a FRB bottle containing PFAS free water and one empty FRB bottle. In the field, pour the water from the one bottle into the empty FRB bottle and label appropriately.
- Request appropriate data deliverable (Category B) and an electronic data deliverable
- For sampling events where multiple private wells (homes or sites) are to be sampled per day, it is acceptable to collect QC samples at a rate of one per 20 across multiple sites or days.

Documentation

A sample log shall document the location of the private well, sample point location, owner contact information, sampling equipment, purge duration, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate and available (e.g. well construction, pump type and location, yield, installation date). Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appendix F - Sampling Protocols for PFAS in Fish

This appendix contains a copy of the current SOP developed by the Division of Fish and Wildlife (DFW) entitled "General Fish Handling Procedures for Contaminant Analysis" (Ver. 8). This SOP should be followed when collecting fish for contaminant analysis. Note, however, that the Bureau of Ecosystem Health will not be supplying bags or tags. All supplies are the responsibility of the collector

Procedure Name: General Fish Handling Procedures for Contaminant Analysis

Number: FW-005

Purpose: This procedure describes data collection, fish processing and delivery of fish collected for contaminant monitoring. It contains the chain of custody and collection record forms that should be used for the collections.

Organization: Environmental Monitoring Section
Bureau of Ecosystem Health
Division of Fish and Wildlife (DFW)
New York State Department of Environmental Conservation (NYSDEC)
625 Broadway
Albany, New York 12233-4756

Version: 8

Previous Version Date: 21 March 2018

Summary of Changes to this Version: Updated bureau name to Bureau of Ecosystem Health. Added direction to list the names of all field crew on the collection record. Minor formatting changes on chain of custody and collection records.

Originator or Revised by: Wayne Richter, Jesse Becker

Date: 26 April 2019

Quality Assurance Officer and Approval Date: Jesse Becker, 26 April 2019

NEW YORK STATE
DEPARTMENT OF ENVIRONMENTAL CONSERVATION

GENERAL FISH HANDLING PROCEDURES FOR CONTAMINANT ANALYSES

A. Original copies of all continuity of evidence (i.e., Chain of Custody) and collection record forms must accompany delivery of fish to the lab. A copy shall be directed to the Project Leader or as appropriate, Wayne Richter. All necessary forms will be supplied by the Bureau of Ecosystem Health. Because some samples may be used in legal cases, it is critical that each section is filled out completely. Each Chain of Custody form has three main sections:

   1. The top box is to be filled out **and signed** by the person responsible for the fish collection (e.g., crew leader, field biologist, researcher). This person is responsible for delivery of the samples to DEC facilities or personnel (e.g., regional office or biologist).

   2. The second section is to be filled out **and signed** by the person responsible for the collections while being stored at DEC, before delivery to the analytical lab. This may be the same person as in (1), but it is still required that they complete the section. Also important is the **range of identification numbers** (i.e., tag numbers) included in the sample batch.

   3. Finally, the bottom box is to record any transfers between DEC personnel and facilities. Each subsequent transfer should be **identified, signed, and dated**, until laboratory personnel take possession of the fish.

B. The following data are required on each **Fish Collection Record** form:

   1. Project and Site Name.

   2. DEC Region.

   3. All personnel (and affiliation) involved in the collection.

   4. Method of collection (gill net, hook and line, etc.)

   5. Preservation Method.

C. The following data are to be taken on each fish collected and recorded on the **Fish Collection Record** form:

   1. Tag number - Each specimen is to be individually jaw tagged at time of collection with a unique number. Make sure the tag is turned out so that the number can be read without opening the bag. Use tags in sequential order. For small fish or composite samples place the tag inside the bag with the samples. The Bureau of Ecosystem Health can supply the tags.

   2. Species identification (please be explicit enough to enable assigning genus and species). Group fish by species when processing.

   3. Date collected.

   4. Sample location (waterway and nearest prominent identifiable landmark).

   5. Total length (nearest mm or smallest sub-unit on measuring instrument) and weight (nearest g or

smallest sub-unit of weight on weighing instrument). Take all measures as soon as possible with calibrated, protected instruments (e.g. from wind and upsets) and prior to freezing.

6. Sex - fish may be cut enough to allow sexing or other internal investigation, but do not eviscerate. Make any incision on the right side of the belly flap or exactly down the midline so that a left-side fillet can be removed.

D. General data collection recommendations:

1. It is helpful to use an ID or tag number that will be unique. It is best to use metal striped bass or other uniquely numbered metal tags. If uniquely numbered tags are unavailable, values based on the region, water body and year are likely to be unique: for example, R7CAY11001 for Region 7, Cayuga Lake, 2011, fish 1. If the fish are just numbered 1 through 20, we have to give them new numbers for our database, making it more difficult to trace your fish to their analytical results and creating an additional possibility for errors.

2. Process and record fish of the same species sequentially. Recording mistakes are less likely when all fish from a species are processed together. Starting with the bigger fish species helps avoid missing an individual.

3. If using Bureau of Ecosystem Health supplied tags or other numbered tags, use tags in sequence so that fish are recorded with sequential Tag Numbers. This makes data entry and login at the lab and use of the data in the future easier and reduces keypunch errors.

4. Record length and weight as soon as possible after collection and before freezing. Other data are recorded in the field upon collection. An age determination of each fish is optional, but if done, it is recorded in the appropriate “Age” column.

5. For composite samples of small fish, record the number of fish in the composite in the Remarks column. Record the length and weight of each individual in a composite. All fish in a composite sample should be of the same species and members of a composite should be visually matched for size.

6. Please submit photocopies of topographic maps or good quality navigation charts indicating sampling locations. GPS coordinates can be entered in the Location column of the collection record form in addition to or instead for providing a map. These records are of immense help to us (and hopefully you) in providing documented location records which are not dependent on memory and/or the same collection crew. In addition, they may be helpful for contaminant source trackdown and remediation/control efforts of the Department.

7. When recording data on fish measurements, it will help to ensure correct data recording for the data recorder to call back the numbers to the person making the measurements.

E. Each fish is to be placed in its own individual plastic bag. For small fish to be analyzed as a composite, put all of the fish for one composite in the same bag but use a separate bag for each composite. It is important to individually bag the fish to avoid difficulties or cross contamination when processing the fish for chemical analysis. Be sure to include the fish’s tag number inside the bag, preferably attached to the fish with the tag number turned out so it can be read. Tie or otherwise secure the bag closed. **The Bureau of Ecosystem Health will supply the bags.** If necessary, food grade bags may be procured from a suitable vendor (e.g., grocery store). It is preferable to redundantly label each bag with a manila tag tied between the knot and the body of the bag. This tag should be labeled with the project name, collection location, tag number, collection date, and fish species. If scales are collected, the scale envelope should be labeled with

the same information.

F. Groups of fish, by species, are to be placed in one large plastic bag per sampling location. **The Bureau of Ecosystem Health will supply the larger bags.** Tie or otherwise secure the bag closed. Label the site bag with a manila tag tied between the knot and the body of the bag. The tag should contain: project, collection location, collection date, species and **tag number ranges**. Having this information on the manila tag enables lab staff to know what is in the bag without opening it.

G. Do not eviscerate, fillet or otherwise dissect the fish unless specifically asked to. If evisceration or dissection is specified, the fish must be cut along the exact midline or on the right side so that the left side fillet can be removed intact at the laboratory. If filleting is specified, the procedure for taking a standard fillet (SOP PREPLAB 4) must be followed, including removing scales.

H. Special procedures for PFAS: Unlike legacy contaminants such as PCBs, which are rarely found in day to day life, PFAS are widely used and frequently encountered. Practices that avoid sample contamination are therefore necessary. While no standard practices have been established for fish, procedures for water quality sampling can provide guidance. The following practices should be used for collections when fish are to be analyzed for PFAS:

No materials containing Teflon.
No Post-it notes.
No ice packs; only water ice or dry ice.
Any gloves worn must be powder free nitrile.
No Gore-Tex or similar materials (Gore-Tex is a PFC with PFOA used in its manufacture).
No stain repellent or waterproof treated clothing; these are likely to contain PFCs.
Avoid plastic materials, other than HDPE, including clipboards and waterproof notebooks.
Wash hands after handling any food containers or packages as these may contain PFCs.
Keep pre-wrapped food containers and wrappers isolated from fish handling.
Wear clothing washed at least six times since purchase.
Wear clothing washed without fabric softener.
Staff should avoid cosmetics, moisturizers, hand creams and similar products on the day of sampling as many of these products contain PFCs (Fujii et al. 2013). Sunscreen or insect repellent should not contain ingredients with “fluor” in their name. Apply any sunscreen or insect repellent well downwind from all materials. Hands must be washed after touching any of these products.

I. All fish must be kept at a temperature <45° F (<8° C) immediately following data processing. As soon as possible, freeze at -20° C ± 5° C. Due to occasional freezer failures, daily freezer temperature logs are required. The freezer should be locked or otherwise secured to maintain chain of custody.

J. In most cases, samples should be delivered to the Analytical Services Unit at the Hale Creek field station. Coordinate delivery with field station staff and send copies of the collection records, continuity of evidence forms and freezer temperature logs to the field station. For samples to be analyzed elsewhere, non-routine collections or other questions, contact Wayne Richter, Bureau of Ecosystem Health, NYSDEC, 625 Broadway, Albany, New York 12233-4756, 518-402-8974, or the project leader about sample transfer. Samples will then be directed to the analytical facility and personnel noted on specific project descriptions.

K. A recommended equipment list is at the end of this document.

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
DIVISION OF FISH AND WILDLIFE
FISH COLLECTION RECORD

Project and Site Name _____ DEC Region _____

Collections made by (include all crew) _____

Sampling Method: □Electrofishing □Gill netting □Trap netting □Trawling □Seining □Angling □Other _____

Preservation Method: □Freezing □Other _____ Notes (SWFDB survey number): _____

FOR LAB USE ONLY- LAB ENTRY NO.	COLLECTION OR TAG NO.	SPECIES	DATE TAKEN	LOCATION	AGE	SEX &/OR REPROD. CONDIT	LENGTH ( )	WEIGHT ( )	REMARKS

richter: revised 2011, 5/7/15, 10/4/16, 3/20/17; becker: 3/23/17, 4/26/19

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
CHAIN OF CUSTODY

I, _____, of _____ collected the
(Print Name) (Print Business Address)

following on _____, 20____ from _____
(Date) (Water Body)

in the vicinity of _____
(Landmark, Village, Road, etc.)

Town of _____, in _____ County.

Item(s) _____

_____

Said sample(s) were in my possession and handled according to standard procedures provided to me prior to collection. The sample(s) were placed in the custody of a representative of the New York State Department of Environmental Conservation on _____, 20_____.

_____ _____
Signature Date

I, _____, received the above mentioned sample(s) on the date specified and assigned identification number(s) _____ to the sample(s). I have recorded pertinent data for the sample(s) on the attached collection records. The sample(s) remained in my custody until subsequently transferred, prepared or shipped at times and on dates as attested to below.

_____ _____
Signature Date

SECOND RECIPIENT (Print Name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
THIRD RECIPIENT (Print Name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
FOURTH RECIPIENT (Print Name)	TIME & DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
RECEIVED IN LABORATORY BY (Print Name)	TIME & DATE	REMARKS
SIGNATURE	UNIT	
LOGGED IN BY (Print Name)	TIME & DATE	ACCESSION NUMBERS
SIGNATURE	UNIT	

richter: revised 21 April 2014; becker: 23 March 2017, 26 April, 2019

NOTICE OF WARRANTY

By signature to the chain of custody (reverse), the signatory warrants that the information provided is truthful and accurate to the best of his/her ability. The signatory affirms that he/she is willing to testify to those facts provided and the circumstances surrounding the same. Nothing in this warranty or chain of custody negates responsibility nor liability of the signatories for the truthfulness and accuracy of the statements provided.

HANDLING INSTRUCTIONS

On day of collection, collector(s) name(s), address(es), date, geographic location of capture (attach a copy of topographic map or navigation chart), species, number kept of each species, and description of capture vicinity (proper noun, if possible) along with name of Town and County must be indicated on reverse.

Retain organisms in manila tagged plastic bags to avoid mixing capture locations. Note appropriate information on each bag tag.

Keep samples as cool as possible. Put on ice if fish cannot be frozen within 12 hours. If fish are held more than 24 hours without freezing, they will not be retained or analyzed.

Initial recipient (either DEC or designated agent) of samples from collector(s) is responsible for obtaining and recording information on the collection record forms which will accompany the chain of custody. This person will seal the container using packing tape and writing his signature, the time and the date across the tape onto the container with indelible marker. Any time a seal is broken, for whatever purpose, the incident must be recorded on the Chain of Custody (reason, time, and date) in the purpose of transfer block. Container then is resealed using new tape and rewriting signature, with time and date.

EQUIPMENT LIST

Scale or balance of appropriate capacity for the fish to be collected.

Fish measuring board.

Plastic bags of an appropriate size for the fish to be collected and for site bags.

Individually numbered metal tags for fish.

Manila tags to label bags.

Small envelops, approximately 2” x 3.5”, if fish scales are to be collected.

Knife for removing scales.

Chain of custody and fish collection forms.

Clipboard.

Pens or markers.

Paper towels.

Dish soap and brush.

Bucket.

Cooler.

Ice.

Duct tape.

Appendix G – PFAS Analyte List

Group	Chemical Name	Abbreviation	CAS Number
Perfluoroalkyl sulfonic acids	Perfluorobutanesulfonic acid	PFBS	375-73-5
	Perfluoropentanesulfonic acid	PFPeS	2706-91-4
	Perfluorohexanesulfonic acid	PFHxS	355-46-4
	Perfluoroheptanesulfonic acid	PFHpS	375-92-8
	Perfluorooctanesulfonic acid	PFOS	1763-23-1
	Perfluorononanesulfonic acid	PFNS	68259-12-1
	Perfluorodecanesulfonic acid	PFDS	335-77-3
	Perfluorododecanesulfonic acid	PFDoS	79780-39-5
Perfluoroalkyl carboxylic acids	Perfluorobutanoic acid	PFBA	375-22-4
	Perfluoropentanoic acid	PFPeA	2706-90-3
	Perfluorohexanoic acid	PFHxA	307-24-4
	Perfluoroheptanoic acid	PFHpA	375-85-9
	Perfluorooctanoic acid	PFOA	335-67-1
	Perfluorononanoic acid	PFNA	375-95-1
	Perfluorodecanoic acid	PFDA	335-76-2
	Perfluoroundecanoic acid	PFUnA	2058-94-8
	Perfluorododecanoic acid	PFDoA	307-55-1
	Perfluorotridecanoic acid	PFTrDA	72629-94-8
	Perfluorotetradecanoic acid	PFTeDA	376-06-7
Per- and Polyfluoroether carboxylic acids	Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6
	4,8-Dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4
	Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
	Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
	Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6
Fluorotelomer sulfonic acids	4:2 Fluorotelomer sulfonic acid	4:2-FTS	757124-72-4
	6:2 Fluorotelomer sulfonic acid	6:2-FTS	27619-97-2
	8:2 Fluorotelomer sulfonic acid	8:2-FTS	39108-34-4
Fluorotelomer carboxylic acids	3:3 Fluorotelomer carboxylic acid	3:3 FTCA	356-02-5
	5:3 Fluorotelomer carboxylic acid	5:3 FTCA	914637-49-3
	7:3 Fluorotelomer carboxylic acid	7:3 FTCA	812-70-4
Perfluorooctane sulfonamides	Perfluorooctane sulfonamide	PFOSA	754-91-6
	N-methylperfluorooctane sulfonamide	NMeFOSA	31506-32-8
	N-ethylperfluorooctane sulfonamide	NEtFOSA	4151-50-2
Perfluorooctane sulfonamidoacetic acids	N-methylperfluorooctane sulfonamidoacetic acid	N-MeFOSAA	2355-31-9
	N-ethylperfluorooctane sulfonamidoacetic acid	N-EtFOSAA	2991-50-6
Perfluorooctane sulfonamide ethanols	N-methylperfluorooctane sulfonamidoethanol	MeFOSE	24448-09-7
	N-ethylperfluorooctane sulfonamidoethanol	EtFOSE	1691-99-2

Group	Chemical Name	Abbreviation	CAS Number
Ether sulfonic acids	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (F-53B Major)	9Cl-PF3ONS	756426-58-1
	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor)	11Cl-PF3OUdS	763051-92-9
	Perfluoro(2-ethoxyethane) sulfonic acid	PFEESA	113507-82-7

Appendix H - Data Review Guidelines for Analysis of PFAS in Non-Potable Water and Solids

General

These guidelines are intended to be used for the validation of PFAS using EPA Method 1633 for projects within the Division of Environmental Remediation (DER). Data reviewers should understand the methodology and techniques utilized in the analysis. Consultation with the end user of the data may be necessary to assist in determining data usability based on the data quality objectives in the Quality Assurance Project Plan. A familiarity with the laboratory's Standard Operating Procedure may also be needed to fully evaluate the data. If you have any questions, please contact DER's Quality Assurance Officer, Dana Barbarossa, at dana.barbarossa@dec.ny.gov.

Preservation and Holding Time

Samples should be preserved with ice to a temperature of less than 6°C upon arrival at the lab. The holding time is 28 days to extraction for aqueous and solid samples. The time from extraction to analysis for aqueous samples is 28 days and 40 days for solids.

Temperature greatly exceeds 6ºC upon arrival at the lab*	Use professional judgement to qualify detects and non-detects as estimated or rejected
Holding time exceeding 28 days to extraction	Use professional judgement to qualify detects and non-detects as estimated or rejected if holding time is grossly exceeded

*Samples that are delivered to the lab immediately after sampling may not meet the thermal preservation guidelines. Samples are considered acceptable if they arrive on ice or an attempt to chill the samples is observed.

Initial Calibration

The initial calibration should contain a minimum of six standards for linear fit and six standards for a quadratic fit. The relative standard deviation (RSD) for a quadratic fit calibration should be less than 20%.

The low-level calibration standard should be within 50% - 150% of the true value, and the mid-level calibration standard within 70% - 130% of the true value.

%RSD >20%	J flag detects and UJ non detects

Continuing Calibration Verification

Continuing calibration verification (CCV) checks should be analyzed at a frequency of one per ten field samples. If CCV recovery is very low, where detection of the analyte could be in question, ensure a low level CCV was analyzed and use to determine data quality.

CCV recovery <70 or >130%	J flag results

Blanks

There should be no detections in the method blanks above the reporting limits. Equipment blanks, field blanks, rinse blanks etc. should be evaluated in the same manner as method blanks. Use the most contaminated blank to evaluate the sample results.

Blank Result	Sample Result	Qualification
Any detection	<Reporting limit	Qualify as ND at reporting limit
Any detection	>Reporting Limit and >10x the blank result	No qualification
>Reporting limit	>Reporting limit and <10x blank result	J+ biased high

Field Duplicates

A blind field duplicate should be collected at rate of one per twenty samples. The relative percent difference (RPD) should be less than 30% for analyte concentrations greater than two times the reporting limit. Use the higher result for final reporting.

RPD >30%	Apply J qualifier to parent sample

Lab Control Spike

Lab control spikes should be analyzed with each extraction batch or one for every twenty samples. In the absence of lab derived criteria, use 70% - 130% recovery criteria to evaluate the data.

Recovery <70% or >130% (lab derived criteria can also be used)	Apply J qualifier to detects and UJ qualifier to non detects

Matrix Spike/Matrix Spike Duplicate

One matrix spike and matrix spike duplicate should be collected at a rate of one per twenty samples. Use professional judgement to reject results based on out of control MS/MSD recoveries.

Recovery <70% or >130% (lab derived criteria can also be used)	Apply J qualifier to detects and UJ qualifier to non detects of parent sample only
RPD >30%	Apply J qualifier to detects and UJ qualifier to non detects of parent sample only

Extracted Internal Standards (Isotope Dilution Analytes)

Problematic analytes (e.g. PFBA, PFPeA, fluorotelomer sulfonates) can have wider recoveries without qualification. Qualify corresponding native compounds with a J flag if outside of the range.

Recovery <50% or >150%	Apply J qualifier
Recovery <25% or >150% for poor responding analytes	Apply J qualifier
Isotope Dilution Analyte (IDA) Recovery <10%	Reject results

Signal to Noise Ratio

The signal to noise ratio for the quantifier ion should be at least 3:1. If the ratio is less than 3:1, the peak is discernable from the baseline noise and symmetrical, the result can be reported. If the peak appears to be baseline noise and/or the shape is irregular, qualify the result as tentatively identified.

Reporting Limits

If project-specific reporting limits were not met, please indicate that in the report along with the reason (e.g. over dilution, dilution for non-target analytes, high sediment in aqueous samples).

Peak Integrations

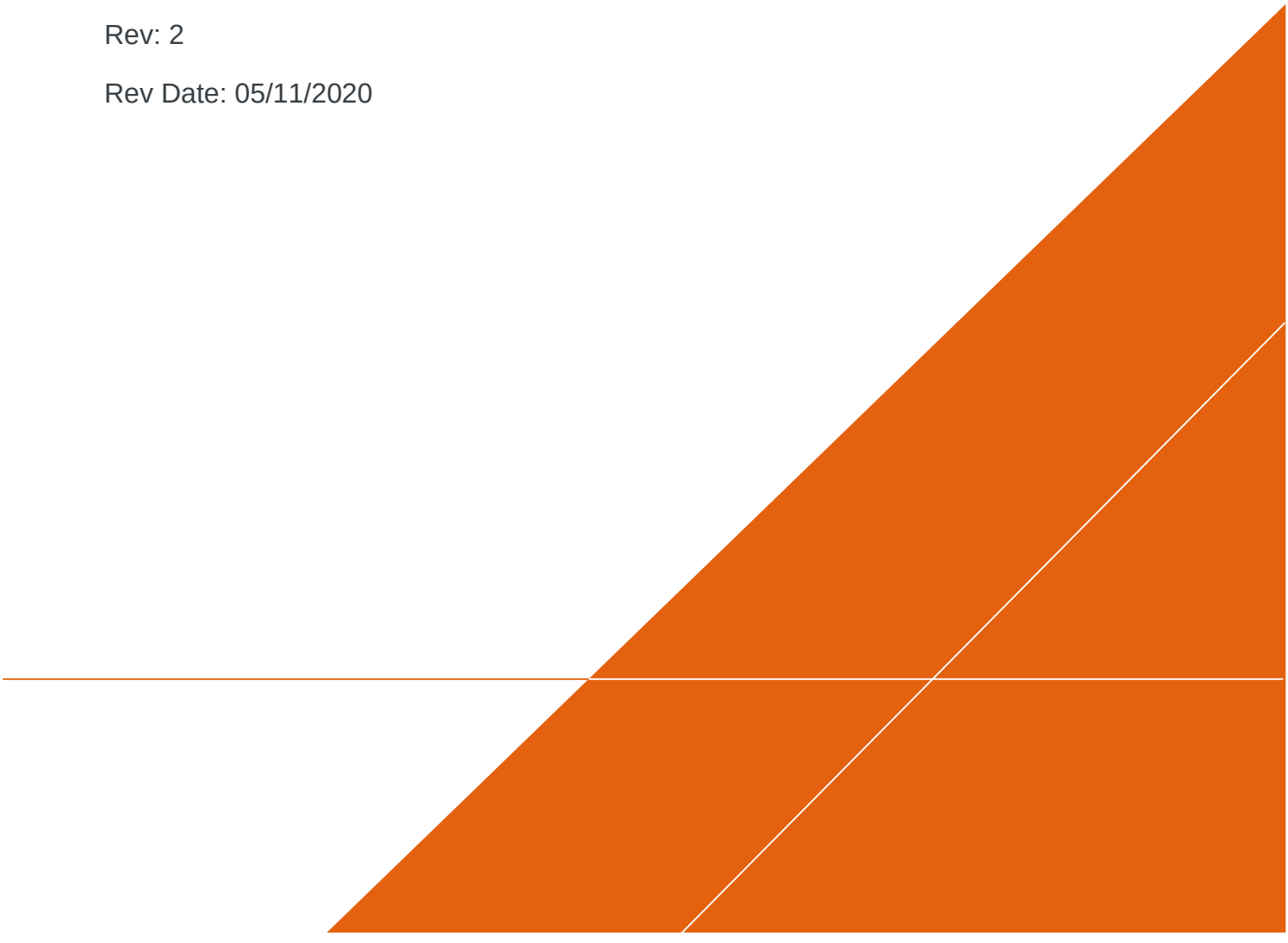
Target analyte peaks should be integrated properly and consistently when compared to standards. Ensure branched isomer peaks are included for PFAS where standards are available. Inconsistencies should be brought to the attention of the laboratory or identified in the data review summary report.

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TGI - EXTRACTION/ PRESERVATION OF SOIL/SEDIMENT FOR VOCS

Rev: 2

Rev Date: 05/11/2020

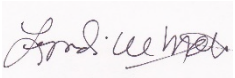


VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
2	May 11, 2020	All	Conversion from SOP to TGI	Lyndi Mott/ Dennis Capria

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APPROVAL SIGNATURES

Prepared by:		04/27/2020
	Lyndi Mott	Date:
Technical Expert Reviewed by:		05/11/2020
	Dennis Capria (Technical Expert)	Date:

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1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

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In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

Soil or sediment samples collected for volatile organic compound (VOC) analysis must be handled in a manner which will minimize the loss of contaminants due to volatilization and biodegradation. Based on experience and open literature, it has been concluded that field extraction and preservation must be conducted in a manner to ensure that contaminants do not degrade or volatilize during sample handling and transport. The following equipment and procedures summarize the method of field preservation of soil samples.

3 PERSONNEL QUALIFICATIONS

Arcadis field personnel will have current health and safety training, including 40-hour HAZWOPER training, site supervision training, and site-specific health and safety training. At least one person on the sampling team must be trained per Department of Transportation (DOT) and (International Air Transportation Administration (IATA) requirements to prepare and offer shipments of samples by a

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commercial carrier and training in Materials of Trade when transporting this material in Arcadis or private vehicles for work-related purposes. Trained personnel will use the following shipping guides:

- Arcadis Hazardous Materials (aka Dangerous Goods) using Shipping Guide No. US-002 - Environmental Samples-Solids Known or Suspected of Being Hazardous per DOT Definition
- Arcadis Hazardous Materials (aka Dangerous Goods) using Shipping Guide No. US-012 – Environmental Samples solid containing Methanol and or Sodium Bisulfate Preservative (collected using TeraCore® Encore®, EazyDraw® Syringe Samplers).

In addition, Arcadis personnel overseeing, directing, or supervising soil collection will be versed in the applicable Quality Procedures (QPs), standard operating procedures (SOPs), and Technical Guidance Instructions (TGIs) to successfully complete the sample activities.

4 EQUIPMENT LIST

- portable balance, small electronic or manual scale calibrated with an appropriate certified weight;
- analyte-free water;
- Site specific Health and Safety Plan (HASP);
- personal protective equipment (PPE), per the HASP;
- 1¾ cm inside-diameter disposable soil coring device syringes (supplied by laboratory);
- coolers or transport containers with contained ice;
- large soil sampling device (e.g., split-spoon sampler);
- indelible ink pens;
- field logbook;
- duct tape;
- 1-gallon freezer bags;
- appropriate forms;
- preservative-free empty sample containers;
- EnCore™ (or equivalent, e.g. TerraCore®, EasyDraw®) samplers and cut plastic syringes; and/or sampling containers provided by the laboratory (dependent on the type of purge and trap unit that will be used to analyze the samples):
    - low-concentration containers with a magnetic stir bar and a solution of 1 gram of sodium bisulfate dissolved in 5 milliliter (mL) of organic-free water. Tare weight of container and contents should be recorded on the label.
    - high-concentration [greater than 200 micrograms per kilogram (ug/kg)] containers will contain 10 mL of methanol and also have the tare weight recorded on the label. Tare weight of the container and contents should be recorded on the label.

5 CAUTIONS

Once the proper weight has been contained within the syringe, the piston of the syringe is used to push the soil into the 40-mL sample vial. Care must be taken so as not to spill or splash the preservative already in the vials. Caution must also be used when recapping the vial, as even a small amount of soil on the rim of the vial may cause improper sealing and subsequently lead to loss of the preservative and

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surrogates. The samples must be placed in coolers and maintained at approximately 4°C. The soil sample vials will be weighed by the laboratory before extractions are completed. For this reason, extra labels or tape are not to be added to the vials. Containers or syringes that differ from the tare weight by more than 0.01 gram should not be used.

Avoid over tightening sample vials lids. Over tightened lids may damage the Teflon seal and integrity of the sample.

Samples unpreserved at 4°C have a holding time of 48-hours, from the time of collection and analysis or preservation. If samples are to be shipped unpreserved, plan to express mail via air and to ship on the same day as collection, unless performance data can be provided to support longer holding times. The 40 mL vials are unacceptable as unpreserved sample containers due to VOC loss via volatilization and biodegradation.

Freezing unpreserved samples in proper containers may be considered to extend holding times to 14 days from collection.

Soils are to be collected and contained in the least amount of time possible to minimize the loss of VOCs. Trimming of outer layer of the exposed the soil sample should be considered, if the sample has been exposed to the atmosphere for more than a couple of minutes.

6 HEALTH AND SAFETY CONSIDERATIONS

Care must be taken so as not to spill or splash the preservative already in the vials, when applicable. Sodium bisulfate is a strong acid and can cause severe burns. If the preservative makes contact, immediately flush with potable water. Immediately consult with a medical professional if any burning, pain, or irritation persists.

The 40-mL sample vials can shatter while tightening. Amber vials are more prone to breakage. Use appropriate cut resistant gloves to avoid possible laceration while tightening vials.

Refer to the site-specific health and safety plan (HASP) for further health and safety considerations.

7 PROCEDURE

Container Preparation

Container preservation may be done either in the field or in the laboratory. Procedures for both methods are provided in this SOP. Standard sample containers for laboratory preservation will include either TerraCore® EnCore®, or EasyDraw®.

Field preservation will depend on the soil concentration, if known.

For high concentration soils (greater than 200 ug/kg), laboratory-prepared 40-mL VOA vials filled with 5 mL to 10 mL of analyte-free purge and trap grade methanol (at a minimum of 5 mL methanol) to 5-gram soil/sediment. The sample container size may be dependent on the type of purge and trap unit that will be used to analyze the samples.

Containers for low VOC concentrations will include 1 gram of sodium bisulfate dissolved in 5 mL of organic-free water.

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The analytical laboratory also adds the appropriate surrogate compounds to the methanol based on the analytical method quality assurance [e.g. 8015, 8021, 8260b, Massachusetts Compendium of Analytical Methods for Volatile Petroleum Hydrocarbons (VPH), or 8260b, USEPA, 2002; DEQE, 2000; MADEP, 2003]. The laboratory records the weight of each vial to the nearest 0.1 gram after both methanol and surrogates have been added. These containers must be stored in coolers and maintained at less than 6°C but not without freezing.

Sampling

Selection of a soil/sediment sampling interval will be determined based on a site-specific sampling plan and collected either via TerraCore® EnCore®, EasyDraw® or by preservation in the field with methanol and/or sodium bisulfate in containers pre-measured by the laboratory.

Preservation in the Field (laboratory pre-filled sample containers)

Soil/sediment is collected in-situ or from a larger sampling device (e.g., split-spoon, auger, or other sampling device) with a disposable coring device (syringe), also supplied by the laboratory, as soon as possible upon sample retrieval to avoid exposure to air and VOC loss. A coring device that may be used of amendable to soil/sediment type consists of a plastic syringe with the tip removed. It is essential that the diameter be suitable for injection into the sample container. A small electronic or manual balance is needed for weighing the syringe and soil.

1. Once the tare weight of the syringe is determined and a sample interval is selected, soil sample collection is accomplished by manually pushing a syringe into the soil/sediment with the piston withdrawn or collection with a stainless-steel spoon.

2. The soil sample weight is determined by subtracting the tared weight from the total weight of the soil and syringe.

3. Acceptable soil sample weight is 5 grams for a 40-mL vial sample container. However, the laboratory may supply larger sized sample containers and require a laboratory-specific weight of soil/sediment.

    - If a syringe is used and the sample weight is less than 5 grams, the syringe may be pushed into the soil again, with the piston withdrawn to gather additional soil.

    - If the sample weight is greater than 5 grams, a portion of soil may be extruded and removed from the syringe.

    - Weigh the containers in the field. Record the weight of each sample. Subtract the tare weight of the container and preservative to obtain the weight of sample. Record the weights on the sample labels and in the field notebook.

    - If the volatile concentrations of the sample are not known, two low-concentration vials and one high-concentration vial should be prepared. Additional vials are needed for samples to be used for laboratory quality control purposes.

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- Note: Samples that contain carbonate minerals may effervesce upon contact with the acidic low-concentration preservative. If the effervescence is small, the loss of volatiles will be limited by quickly capping the vial. If large amounts of gas are generated, target analytes may be lost, and the vials may shatter. If this occurs, document the issue in the field notebook and collect another sample using a fresh container prepared with 5 mL (1 mL of water is equivalent to 1 gram of water) of analyte-free water and no preservative.

- Place samples on ice immediately after collection and ship or deliver to the laboratory as soon as possible.

- An additional sample fraction should be placed in a clean glass jar with no preservative for laboratory use to determine percent solids. Moisture content determination is required to report the sample results on a dry weight basis. Moisture content determination is required to report the sample results on a dry weight basis. If soil samples will be analyzed for other parameters, the moisture content can usually be taken from the other sample containers. However, when sampling for VOCs only, a small separate laboratory-supplied container must be filled.

Laboratory Preservation (Encore™ or equivalent)

1. Collect an approximate 5-gram sample using an EnCore™ sampler or cut plastic syringe.

2. Place samples on ice immediately after collection and ship or deliver to the laboratory as soon as possible. Samples in capped EnCore™ samplers should be delivered within 24 hours of collection to allow the sample to be preserved within 48 hours from collection and meet holding time.

3. In addition to the soil sample collected for VOC analysis, soil must be collected and placed into an empty container for moisture content analysis. Moisture content determination is required to report the sample results on a dry weight basis. If soil samples will be analyzed for other parameters, the moisture content can usually be taken from the other sample containers. However, when sampling for VOCs only, a small separate laboratory-supplied container must be filled.

Shipping Container Preparation

Use Arcadis Hazardous Materials (aka Dangerous Goods) using Shipping Guide No. US-012 – Environmental Samples solid containing Methanol and or Sodium Bisulfate Preservative (collected using TerraCore® EnCore®, EasyDraw® Syringe Samplers) for sample container preparation. Among other requirements depending on whether the samples are shipped by air verse by ground shipping containers must be marked with **"This package conforms to 49 CFR 173.4."** Note: in order to comply with 49 CFR 173.4, the total volume of methanol mixture per shipping container must be less than 30 grams. In addition, other pertinent requirements of this reference are as follows:

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- Each inner receptacle is securely packed in an inside packaging with cushioning and absorbent material. The inside packaging cannot react chemically with the material and needs to be capable of absorbing the entire contents of the receptacle. (Note: a foam container for the vials meets these requirements.)

- The inside packaging is securely packed in a strong outside packaging. (Note: a cooler meets this requirement.)

- The gross mass of the completed package does not exceed 64 pounds.

Consult Arcadis Hazardous Materials (aka Dangerous Goods) using Shipping Guide No. US-012 – Environmental Samples, Solid Containing Methanol and/or Sodium Bisulfate Preservative (collected using TerraCore® EnCore®, EasyDraw® Syringe Samplers) for appropriate packing and labeling for shipments via air and ground. In addition, consult the Chain-of-Custody SOP.

8 WASTE MANAGEMENT

Used methanol or sodium bisulfate, if any, will be contained in an airtight drum or container. Containers will be labeled with the project name, date, and contents. Appropriate client specified personnel will be notified for the transport of container to the appropriate on-site storage area for disposal at an appropriate facility. Any unused sample containers with methanol or sodium bisulfate will be shipped back to the laboratory using original packaging material per DOT or IATA procedures.

9 DATA RECORDING AND MANAGEMENT

Sampling activities, (i.e. location, depth, soil/sediment type, sample identification, sample container tare weight, sample weights, effervescence) will be recorded in the field logbook. Chain of custody (COC) will be prepared per the approved SOP and copies of the COC will be transmitted to the project manager and maintained in project files.

10 QUALITY ASSURANCE

Quality assurance activities will be completed to comply with the site-specific sampling plan and/or quality assurance project plan.

A methanol trip blank should accompany the sample containers at all times in the field and during transport. This consists of a sample container prepared in a similar manner as the soil sample containers. One trip blank should accompany each sample delivery group.

Field blanks and ambient air blanks are optional when sampling via the methanol preservation method.

Discrete blind duplicate samples may be collected in separate soil/sediment sample containers. Compositing or homogenizing multiple soil/sediment samples into one sample container is not acceptable for VOCs.

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Site-specific matrix spike and matrix spike duplicate (MS/MSD) analyses, if required, may be collected from a single soil/sediment sample container; no additional sample volume is required

11 REFERENCES

Arizona Department of Environmental Quality (ADEQ), 2000. Implementation of EPA Method 5035-Soil Preparation of EPA Method 8015B, 8121B and 8260B, Arizona Revised Statues (A.R.S.) 49-104 (A). April 19, 2000.

Massachusetts Department of Environmental Protection (MADEP), 2003. Method for the Determination of Volatile Petroleum Hydrocarbons, MADEP-VPH-03-1.1, Revision 1.1. December 2003.

USEPA, 2002. *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition, Method 5035A – Closed System Purge and Trap for Volatile Organics in Soils and Waste Samples. July 2002.

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TGI – Soil Description

Rev: 4

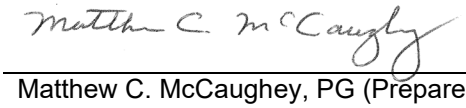
Rev Date: June 14, 2022

Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
	0	May 20, 2008	17	Original SOP	Joe Quinnan Joel Hunt
	1	September 2016	15	Updated to TGI	Nick Welty Patrick Curry
	2	February 16, 2018	15	Updated descriptions, attachments and references in text	Nick Welty Patrick Curry
	3	April 15, 2022		Minor description edits, intro of grain-size K analysis, revised boring log template	Matt McCaughey Patrick Curry
	4	June 14, 2022		Updated date on cover page and header. Updated revision number from 3 to 4. Updated reference throughout document from ASTM D2488-06 to ASTM D-2488.17 . Change “25% silt and clay; 15% pebbles” to “20% silt and 20% clay” on page 10 of 23. Updates made to Section 8.2.1 Changed reference to Appendix B to Appendix A	

Approval Signatures

Prepared by: 6/14/2022



Matthew C. McCaughey, PG (Preparer) Date

Reviewed by: 6/14/2022



Patrick Curry, PG (Subject Matter Expert) Date

1 Introduction

This Arcadis Technical Guidance Instruction (TGI) describes proper soil description procedures based on visual inspection and testing of soil cores and samples. This document has been developed to emphasize field observation and documentation of details required to:

- Make hydrostratigraphic interpretations guided by depositional environment/geologic settings
- Provide information needed to understand the distribution of constituents of concern; properly design wells, piezometers, and/or additional field investigations; and develop appropriate remedial strategies.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

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3 Scope and Application

This TGI should be followed for unconsolidated material unless there is an established client-required specific procedure or regulatory-required specific procedure. In cases where there is a required specific procedure, it should be followed and should be referenced and/or provided as an appendix to reports that include soil classifications and/or boring logs. When following a required non-Arcadis procedure, additional information required by this TGI should be included in field notes with client approval.

This TGI incorporates elements from various standard systems such as ASTM D-2488-17, Unified Soil Classification System, Burmister and Udden Wentworth. However, none of these standard systems focus specifically on contaminant hydrogeology and remedial design. Therefore, although each of these systems contain valuable guidance and information related to correct descriptions, strict application of these systems can omit information critical to our clients and the projects that we perform.

This TGI includes the following attachments:

- **Attachment A –** Field Soil Description Guide
- **Attachment B –** Particle Size System Comparison
- **Attachment C –** Description of Logging Terms
- **Attachment D –** Blank Boring Log
- **Attachment E –** Completed Boring Log

This TGI does not address details of health and safety; drilling method selection; boring log preparation; sample collection; or laboratory analysis. Refer to other Arcadis procedure, guidance, and instructional documents, the project work plans including the quality assurance project plan, sampling plan, and health and safety plan (HASP), as appropriate.

4 Personnel Qualifications

Soil descriptions should only be performed by Arcadis personnel or authorized sub-contractors with a degree in geology or a geology-related discipline. Field personnel will complete training on the Arcadis soil description TGI in the office and/or in the field under the guidance of an experienced field geologist with at least 2 years of prior experience applying the Arcadis soil description method.

5 Equipment List

The following equipment should be taken to the field to facilitate soil descriptions:

- Field book, field forms or digital devices to record soil descriptions
- Field book for supplemental notes
- This TGI for Soil Descriptions and any project-specific procedure, guidance, and/or instructional documents (if required)
- Field card showing Wentworth scale
- Munsell® soil color chart
- Tape measure divided into tenths of a foot
- Stainless steel knife or spatula
- Hand lens
- Water squirt bottle
- 4-ounce glass jars with lids (for collecting soil core samples)
- Personal protective equipment (PPE), as required by the HASP
- Digital camera

- Folding table

6 Cautions

Drilling and drilling-related hazards including subsurface utilities are discussed in other procedure documents and site-specific HASPs and are not discussed herein.

Soil samples may contain hazardous substances that can result in exposure to persons describing soils. Routes for exposure may include dermal contact, inhalation and ingestion. Refer to the project specific HASP for guidance in these situations.

7 Health and Safety Considerations

Field activities associated with soil sampling and description will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities. Know what hazardous substances may be present in the soil and understand their hazards. Always avoid the temptation to touch soils with bare hands, detect odors by placing soils close to your nose, or tasting soils.

8 Procedure

8.1 General Procedures

- Select the appropriate sampling method to obtain representative samples in accordance with the selected sub-surface exploration method, e.g., split-spoon or Shelby sample for hollow-stem drilling, acetate sleeves for direct push, bagged core for sonic drilling, etc.
- Proceed with field activities in required sequence. Although completion of soil descriptions is often not the first activity after opening sampler, identification of stratigraphic changes is often necessary to select appropriate intervals for field screening and/or selection of laboratory samples.
- Set up boring log field sheet.
    - Determine the proper units of measure. Drillers in both the US and Canada generally work in feet due to equipment specifications. Field geologists typically record drilling depths, core recovery, and sample intervals in feet and grain size in millimeters
    - Use the Arcadis standard boring log form (**Attachment D**). *Note that as of April 2022, several digital logging applications are available through the FieldNow™ program and the Fulcrum app. A future revision of this TGI, likely in early 2023, will emphasize digital logging methods and field boring log forms will no longer be acceptable. FieldNow is discussed further in Section 10.*
    - The boring log template includes a graphic log of the primary soil texture to support quick visual evaluation of grain size. The purpose of the graphic log is to quickly assess relative soil permeability. Note, for poorly sorted soils (e.g., glacial till), the principal component may not correlate to permeability of the sample. In this case, the geologist should use best judgement to graph overall soil type consistent with relative soil permeability. For example, for a dense sand/silt/clay till, the graphic log would reflect the silt/clay, rather than sand.

    - Record depths along the left-hand side at a standard scale to aid in the use of this tool.
- Examine each soil core (this is different than examining each sample selected for laboratory analysis) and record the soil conditions in accordance with guidelines provided in Section 8.2.
- At the end of the boring, record the amount of drilling fluid used (if applicable) and the total depth logged.
- At a minimum, a written or digital boring log should be prepared with the following information:
    - Describe type of surface material (asphalt, grass, topsoil, gravel, etc.)
    - Describe the type of fill or non-native soils and estimated depth to native soils
    - Record sample intervals (soil cores, environmental and/or geotechnical samples)
    - Describe soil conditions in accordance with this TGI
    - Record moisture content and estimated depth to water table or saturated zone
    - Record the total depth and document why drilling was stopped (refusal, target depth achieved, etc.)

8.2 Soil Description Procedures

The standard soil description order is presented below.

- Depth
- PRIMARY TEXTURE
- Principal and Minor Components with Descriptors
    - % Modifiers and grain size fraction
    - Angularity for coarse sand and larger particles
    - Consistency or Density
    - Plasticity for silt and clay
    - Dilatancy for silt and silt-sand mixtures
- Sorting
- Moisture Content
- Color
- Notes

Depth. To measure and record the depth below ground surface (bgs) of top and bottom of each stratum, the following information should be recorded.

- Measured depth to the top and bottom of sampled interval. Use starting depth of sample based upon measured tool length information and the length of sample interval.
- Length of sample recovered, not including slough (material that has fallen into hole from previous interval), expressed as fraction with length of recovered sample as numerator over length of sampled interval as denominator (e.g., 36/60 for 36 inches recovered from 5-ft [60-inch] sampling interval).
- Thickness of each stratum measured sequentially from the top of recovery to the bottom of recovery.
- Any observations of sample condition or drilling activity that would help identify whether there was loss from the top of the sampling interval, loss from the bottom of the sampling interval, or compression of the sampling interval. Examples: 14/24, gravel in nose of spoon; or 36/60 bottom 12 inches of core empty.

Determination of Components. Obtain a representative sample of soil from a single stratum. If multiple strata are present in a single sample interval, each stratum should be described separately. More specifically, if the sample is from a 2-foot-long split-spoon where strata of coarse sand, fine sand and clay are present, then the resultant description should be of the three individual strata unless a combined description can clearly describe the interbedded nature of the three strata. Example: SAND, fine; with interbedded lenses of Silt and Clay, ranging between 1 and 3 inches thick.

Identify principal component and express volume estimates for minor components on logs using the following standard modifiers.

Modifier	Percent of Total Sample (by volume)
and	36 – 50
some	21 - 35
little	10 - 20
trace	<10

Determination of components is based on using the Udden-Wentworth particle size classification (see below) and measurement of the average grain size diameter. Each size class differs from the next larger class by a constant ratio of ½. Due to visual limitations, the finer classifications of Wentworth's scale cannot be distinguished in the field and the subgroups are not included. Visual determinations in the field should be made carefully by comparing the sample to the Soil Description Field Guide (**Attachment A**) that shows Udden-Wentworth scale or by measuring with a ruler.

The following table summarized the modified Udden-Wentworth Scale for grain size classification. Note that gravel is a size category encompassing the granule, pebble, cobble, and boulder size classes.

Udden-Wentworth Scale (Modified by Arcadis, 2008)				
Size Category	**Size Class**	**Millimeters**	**Inches**	**Standard Sieve #**
Gravel (Cobble)	Boulder	256 – 4096	10.08+	
	Large cobble	128 - 256	5.04 -10.08	
	Small cobble	64 - 128	2.52 – 5.04	
Gravel (Pebble)	Very large pebble	32 – 64	0.16 - 2.52	
	Large pebble	16 – 32	0.63 – 1.26	
	Medium pebble	8 – 16	0.31 – 0.63	
	Small pebble	4 – 8	0.16 – 0.31	No. 5 +
	Granule	2 – 4	0.08 – 0.16	No.5 – No.10

Sand	Very coarse sand	1 -2	0.04 – 0.08	No.10 – No.18
	Coarse sand	½ - 1	0.02 – 0.04	No.18 - No.35
	Medium sand	¼ - ½	0.01 – 0.02	No.35 - No.60
	Fine sand	1/8 -¼	0.005 – 0.1	No.60 - No.120
	Very fine sand	1/16 – 1/8	0.002 – 0.005	No. 120 – No. 230
Fines	Silt (subgroups not included)	1/256 – 1/16	0.0002 – 0.002	Not applicable (analyze by pipette or hydrometer)
	Clay (subgroups not included	1/2048 – 1/256	0.00002 – 0.0002	

Identify components as follows. Remove particles greater than very large pebbles (64-mm diameter) from the soil sample. Record the volume estimate of the greater than very large pebbles. Examine the sample fraction of very large pebbles and smaller particles and estimate the volume percentage of the pebbles, granules, sand, silt and clay. Use the jar method, visual method, and/or wash method (Appendix X4 of ASTM D2488) to estimate the volume percentages of each category.

Sieve and hydrometer grain-size analysis can be used to vet the visual description, as well as used to estimate hydraulic conductivity. Lab or field sieve analysis is advisable to characterize the variability and facies trends within each hydrostratigraphic unit. It is recommended that sieve-hydrometer analysis be performed on representative samples from each soil type to estimate the fraction of each grain size category using ASTM D422 Standard Test Method for Particle-Size Analysis of Soils. If desired sieve sizes can be specified to follow the Udden-Wentworth classification (U.S. Standard sieve sizes 6; 12; 20; 40; 70; 140; and 270) to retain pebbles; granules; very coarse sand; coarse sand; medium sand; fine sand; and very fine sand, respectively.

Several empirical formulas provide a reliable means of estimating hydraulic conductivity (K) from grain-size distribution data, provided that the formation does not contain abundant fines that result in cohesive or plastic behavior or include cobble-sized grains (Payne et al. 2008). Grain-size analysis can help bracket the permeability of hydrostratigraphic units (HSUs) and identify order-of-magnitude spatial variations in K. Arcadis has completed modifications to the Excel-based program HydroGeoSieveXL (Devlin 2015) to process sieve data quickly and estimate K. The tool calculates estimated K values from grain-size data using 15 different empirical formulas. A decision matrix then selects which of the formulas is relevant for the soil type and calculates an average K.

Principal Component. The principal component is the size fraction or range of size fractions containing the majority of the volume. Examples: the principal component in a sample that contained 55% small to medium pebbles would be “PEBBLES, small to medium”; or the principal component in a sample that was 20% fine sand, 30% medium sand and 25% coarse sand would be “SAND, fine to coarse” or for a sample that was 40% silt and 45% clay the principal component would be “CLAY and SILT”.

The boring log form (**Appendix D**) includes a graphic log to visually illustrate a relative estimate of soil permeability. To use the graphic log, place an ‘X’ or shade the appropriate column for the primary soil texture. If the soils have a high percentage of a secondary soil texture (i.e., when the ‘and’ modifier’ is used), it’s acceptable to mark off the appropriate column for the secondary soil texture in this instance. However, care should be used to avoid marking off the columns for other minor soil textures because doing so will make it difficult to determine the relative soil permeability of the poorly sorted soils.

As noted above, for poorly sorted soils such as glacial till, the principal component may not correlate to permeability of the sample. In this case, the geologist should use best judgement to graph overall soil type consistent with relative soil permeability.

Minor Component(s). The minor component(s) are the size fraction(s) containing less than 50% volume. Example: the identified components are estimated to be 60% medium sand to granules, 20% silt and 20% clay – there are two identified minor components: silt and clay.

Include a standard modifier to indicate percentage of minor components (see particle size table) and the same descriptors that would be used for a principal component. An example of minor constituents with modifiers include: some silt and clay, low plasticity; little medium to large pebbles, sub-round.

8.2.1 Secondary Descriptors

The following are the descriptors used outside of the principal and minor components. Note that plasticity should be provided as a descriptor for clay and clay mixtures. Dilatancy should be provided for silt and silt mixtures. Angularity should be provided as a descriptor for pebbles and coarse sand.

Angularity. Describe the angularity for coarse sand and larger particles in accordance with the table below (ASTM D-2488-17). Figures showing examples of angularity are available in ASTM D2488-17 and the Arcadis Soil Description Field Guide (**Appendix A**).

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Sub-Angular	Particles are like angular description but have rounded edges
Sub-Rounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges.

Plasticity. Describe the plasticity for silt and clay based on observations made during the following test method (ASTM D-2488-17).

- As in the dilatancy test (described below), select enough material to mold into a ball about ½ inch (12 mm) in diameter. Mold the material, adding water, if necessary, until it has a soft, but not sticky, consistency.
- Shape the test specimen into an elongated pat and roll by hand on a smooth surface or between the palms into a thread about 1/8 inch (3 mm) in diameter. If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation. Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about 1/8 inch. The thread will crumble when the soil is near the plastic limit.

Description	Criteria
Non-plastic	A 1/8-inch (3 mm) thread cannot be rolled at any water content.
Low	The thread can barely be rolled, and the lump cannot be formed when drier than the plastic limit.
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit.

Dilatancy. Describe the dilatancy for silt and silt-sand mixtures using the following field test method (ASTM D-2488-17).

- From the specimen, select enough material to mold into a ball about ½ inch (12 mm) in diameter. Mold the material adding water, if necessary, until it has a soft, but not sticky, consistency.
- Smooth the ball in the palm of one hand with a small spatula.
- Shake horizontally, striking the side of the hand vigorously with the other hand several times.
- Note the reaction of water appearing on the surface of the soil.
- Squeeze the sample by closing the hand or pinching the soil between the fingers, and not the reaction as none, slow, or rapid in accordance with the table below. The reaction is the speed with which water appears while shaking and disappears while squeezing.

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

Note that silt and silt-sand mixtures will be non-plastic and display dilatancy. Clay mixtures will have some degree of plasticity but do not typically react to dilatancy testing. Therefore, the tests outlined above can be used to differentiate between silt-dominated and clay-dominated soils.

Sorting. Sorting is the opposite of grading, which is a commonly used term in the USCS or ASTM methods to describe the uniformity of the particle size distribution in a sample. Well-sorted samples are poorly graded and poorly sorted samples are well graded. Arcadis prefers the use of sorting for particle size distributions and grading to describe particle size distribution trends in the vertical profile of a sample or hydrostratigraphic unit because of

the relationship between sorting and the energy of the depositional process. For soils with sand-sized or larger particles, sorting should be determined as follows:

Description	Criteria
Well Sorted	the range of particle sizes is limited (e.g., the sample is comprised of predominantly one or two grain sizes)
Poorly Sorted	A wide range of particle sizes are present

You can also use sieve analysis to estimate sorting from a sedimentological perspective; sorting is the statistical equivalent of standard deviation. Smaller standard deviations correspond to higher degree of sorting (see Remediation Hydraulics, 2008).

Consistency or Density. This can be determined by standard penetration test (SPT) blow counts (ASTM D-1586) obtained when using hollow-stem auger drilling methods and a split spoon sampling device. Otherwise, some field tests are available as outlined below. When drilling with hollow-stem augers and split-spoon sampling, the SPT blow counts and N-value is used to estimate density. The N-value is the blows per foot for the 6" to 18" interval. For example, for a 24-inch split spoon soil core, the recorded blows per 6-inch interval are: 4/6/9/22. Since the second interval is 6" to 12", the third interval is 12" to 18", the N value is 6+9, or 15. Fifty blow counts for less than 6 inches is considered refusal. In recent years, more common drilling methods include rotary-sonic or direct push. When blow counts are not available, density is determined using a thumb test. Note however, the thumb test only applies to fine-grained soils.

Fine-grained soil – Consistency

Description	Criteria	Blow Counts (6-12 to 12-18-inch split spoon interval)
Very soft	Easily penetrated several inches by thumb	N-value < 2
Soft	Easily penetrated one inch by thumb	N-value 2-4
Medium Stiff	Indented about ½ inch with much effort	N-value 5-8
Stiff	Indented with ¼ inch with great effort	N-value 9-15
Very Stiff	Readily indented by thumbnail	N-value 16-30
Hard	Indented by thumbnail with difficulty	N-value > than 30

Coarse-grained soil – Density

Description	Criteria	Blow Counts (6-12 to 12-18-inch split spoon interval)
Very loose	Density classification of coarse-grained soils is only required when blow counts from standard penetration tests are performed during hollow-stem auger drilling	N-value 1- 4
Loose		N-value 5-10
Medium dense		N-value 11-30
Dense		N-value 31- 50
Very dense		N-value >50

Moisture Content. Moisture content should be described for each soil sample in accordance with the table below (percentages should not be used unless determined in the laboratory). *Note that some drilling methods (e.g., sonic) can compress and dry out the sample during drilling. Therefore, it can be difficult to determine if a sample is saturated, or merely moist. In this case, care should be taken to try and determine a static water level within the borehole by measuring depth to water through the drill casing, if possible.*

Description	Criteria
Dry	Absence of moisture, dry to touch, dusty
Moist	Damp but no visible water
Wet	Visibly free water

Color. Color should be described using simple basic terminology and modifiers based on the Munsell system. Munsell alpha-numeric codes are required for all samples. If the sample contains layers or patches of varying colors this should be noted, and all representative colors should be described. The colors should be described for moist samples. If the sample is dry, it should be wetted prior to comparing the sample to the Munsell chart.

Notes. Additional comments should be made where observed and should be presented as notes with reference to a specific depth interval(s) to which they apply. Some of the significant information that may be observed includes the following.

- Odor - You should not make an effort to smell samples by placing near your nose since this can result in unnecessary exposure to hazardous materials. However, odors should be noted if they are detected during the normal sampling procedures. Odors should be based upon descriptors such as those used in NIOSH “Pocket Guide to Chemical Hazards”, e.g., “pungent” or “sweet” and should not indicate specific chemicals such as “phenol-like” odor or “BTEX” odor.
- Structure
- Bedding planes (laminated, banded, geologic contacts).
- Presence of roots, root holes, organic material, man-made materials, minerals, etc.
- Mineralogy

- Cementation
- NAPL presence/characteristics, including sheen (based on client-specific guidance).
- Reaction with HCl - typically only used for special soil conditions, such as caliche environments.
- Origin, if known (Lacustrine; Fill; etc.).

8.3 Example of Soil Descriptions

The standard generic description order is presented below.

- Depth
- PRIMARY TEXTURE
- Principal and Minor Components with Descriptors
    - % Modifiers and grain size fraction
    - Angularity for coarse sand and larger particles
    - Consistency or Density
    - Plasticity for silt and clay
    - Dilatancy for silt and silt-sand mixtures
- Sorting
- Moisture Content
- Color
- Notes



10-15 feet CLAY, trace silt, trace small to very large pebbles, subround to subangular up to 2" diameter; medium to high plasticity, stiff, moist, dark grayish brown (10YR 4/2). NOTE: Lacustrine; laminated 0.1 to 0.2" thick, laminations brownish yellow (10YR 4/3).



10 -15 feet SAND, medium to very coarse, little granules to medium pebbles, subround to subangular, trace silt; poorly sorted, wet, grayish brown (10YR5/2).

Unlike the first example where a density of cohesive soils could be estimated, this rotary-sonic sand and pebble sample was disturbed during drilling (due to vibrations in a loose sand and pebble matrix) so no density description could be provided. Neither sample had noticeable odor so odor comments were not included.

9 Waste Management

Project-specific requirements should be identified and followed. The following procedures, or similar waste management procedures are generally required.

Water generated during cleaning procedures will be collected and contained onsite in appropriate containers for future analysis and appropriate disposal. PPE (such as gloves, disposable clothing, and other disposable equipment) resulting from personnel cleaning procedures and soil sampling/handling activities will be placed in plastic bags. These bags will be transferred into appropriately labeled 55-gallon drums or a covered roll-off box for appropriate disposal.

Soil materials will be placed in sealed 55-gallon steel drums or covered roll-off boxes and stored in a secured area. Once full, the material will be analyzed to determine the appropriate disposal method.

10 Data Recording and Management

10.1 Digital Data Collection Process Overview

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

10.2 Digital Data Collection Tools for Soil Descriptions

Arcadis is transitioning from the use of paper forms to a digital soil description logging process using web-based FieldNow applications accessible on field tablets and smart phones. Company-wide roll out of a FieldNow application for soil descriptions is targeted by the end of 2022.

Paper forms are included in Revision 3 (April 2022) of this Soil Description TGI. Specifically, a blank boring log and completed boring log are provided in **Attachment D** and **Attachment E**. Additional guidance and examples of the digital data collection tools for soil descriptions will be provided in the next revision to this TGI.

10.3 Additional Guidance

The general logging scheme for soil descriptions is described in this document. Depending on project data quality objectives, specific soil description parameters that are not applicable to project goals may be omitted at the project manager's discretion. In any case, use of consistent procedures is required.

Completed logs and/or logbook will be maintained in the task/project field records file. Digital photographs of typical soil types observed at the site and any unusual features should be obtained whenever possible. Photographs should include a ruler or common object for scale. Photo location, depth and orientation must be recorded in the daily log or logbook and a label showing this information in the photo is useful.

For projects involving soil logging and soil sampling, the soil sample should be recorded on the Arcadis boring log form and the field logbook based on Data Quality Objectives for the task/project.

11 Quality Assurance

Soil descriptions should be completed only by appropriately trained personnel. Descriptions should be reviewed by an experienced field geologist for content, format and consistency. Edited boring logs should be reviewed by the original author to assure that content has not changed.

12 References

ASTM D-1586, Test Method for Penetration Test and Split-Barrel Sampling of Soils.

ASTM D-2488-17, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)

ASTM D422, 63rd Edition, 1972 - Standard Test Method for Particle-Size Analysis of Soils.

Devlin, J.F. 2015. HydroGeoSieve XL: an Excel-based tool to estimate hydraulic conductivity from grain-size analysis. Hydrogeology Journal, DOI 10.1007/s10040-015-1255-0.

Folk, Robert L. 1980. Petrology of Sedimentary Rocks, p. 1-48.

Payne, F. C., Quinnan, J. A., & Potter, S. T. 2008. Remediation Hydraulics. Boca Raton: FL: CRC Press.

United States Bureau of Reclamation. Engineering Geology Field Manual. United States Department of Interior, Bureau of Reclamation. http://www.usbr.gov/pmts/geology/fieldmap.htm.

Munsell® Color Chart – available from Forestry Suppliers, Inc.- Item 77341 “Munsell® Color Soil Color Charts.

Field Gauge Card that Shows Udden-Wentworth scale – available from Forestry Suppliers, Inc. – Item 77332 “Sand Grain Sizing Folder.”

NIOSH Pocket Guide to Chemical Hazards.

Attachment A

Soil Field Reference Guide

The purpose of this attachment is to present a field reference guide for use during soil logging. Field staff are encouraged to bring a laminated copy of this reference guide into the job site.

ON THE ORIGINAL DRAWING.
USE TO VERIFY FIGURE
REPRODUCTION SCALE

SOIL DESCRIPTION FIELD GUIDE (APRIL, 2022; REV. 3.0)

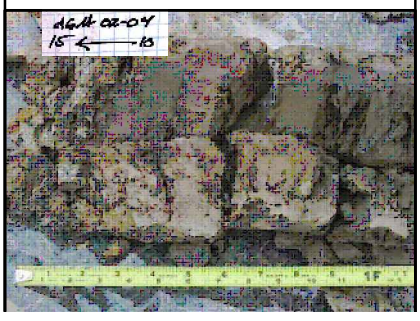
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FINE-GRAINED SOILS

Description	Criteria
	Descriptor - Plasticity
Nonplastic	A 1/8-inch (3 mm) thread cannot be rolled at any water content.
Low	The thread can barely be rolled, and the lump cannot be formed when drier than the plastic limit.
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit.
	Descriptor - Dilatancy
No Dilatancy	No visible change when shaken or squeezed.
Slow	Water appears slowly on the surface of soil during shaking and does not disappear or disappears slowly when squeezed.
Rapid	Water appears quickly on surface of soil during shaking and disappears quickly when squeezed.
Minor Components with Descriptors	
	Moisture
Dry	Absence of moisture, dry to touch, dusty.
Moist	Damp but no visible water.
Wet	Visible free water; soil is usually below the water table. (Saturated)
	Consistency
Very soft	N-value < 2 or easily penetrated several inches by thumb.
Soft	N-value 2-4 or easily penetrated 1 inch by thumb.
Medium stiff	N-value 5-8 or indented about 1/2 inch by thumb with great effort.
Stiff	N-value 9-15 or indented about 1/4 inch by thumb with great effort.
Very stiff	N-value 16-30 or readily indented by thumb nail.
Hard	N-value > than 30 or indented by thumbnail with difficulty.
Color using Munsell	
Geologic Origin (if known)	
Other	

EXAMPLE OF SOIL DESCRIPTION AND PHOTO

10-15 feet CLAY, trace silt, trace small to very large pebbles, subround to subangular up to 2" diameter; medium to high plasticity, stiff, moist, dark grayish brown (10YR 4/2). NOTE: Lacustrine; laminated 0.1 to 0.2" thick, laminations brownish yellow (10YR 4/3).



DESCRIPTION ORDER

Depth Interval
PRIMARY TEXTURE (e.g., SAND)
Principal and Minor Components with Descriptors:
- % Modifiers and grain size fraction
- Angularity coarse sand and larger
- Consistency or Density
- Plasticity for silt and clay
- Dilatancy for silt and silt-sand

Sorting for granular sediments
Moisture Content
Color
Other NOTES

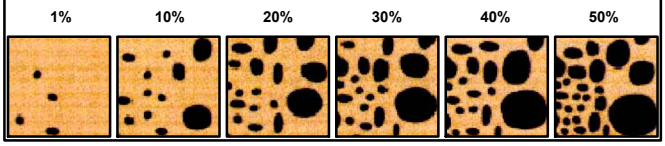
MINOR COMPONENTS % MODIFIERS

Modifier	Percent of Total Sample (by volume)
and	36 - 50
some	21 - 35
little	10 - 20
trace	<10

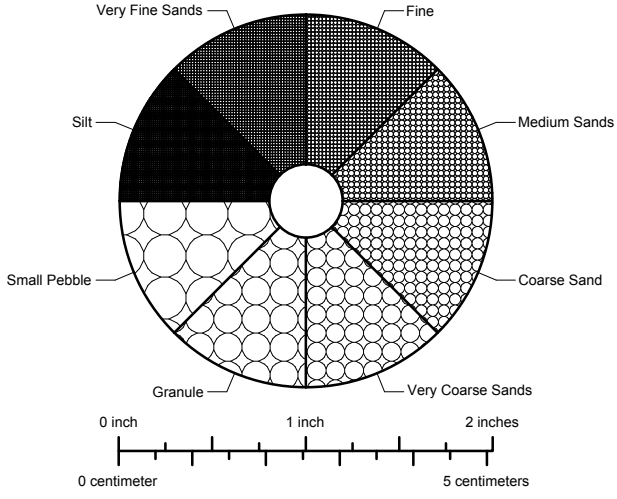
UDDEN-WENTWORTH SCALE

Fraction	Sieve Size	Grain Size	Approximate Scale
Boulder		256 - 4096 mm	Larger than volleyball
Large Cobble		128 - 256 mm	Softball to volleyball
Small Cobble		64 - 128 mm	Pool ball to softball
Very Large Pebble		32 - 64 mm	Pinball to pool ball
Large Pebble		16 - 32 mm	Dime size to pinball
Medium Pebble		8 - 16 mm	Pencil eraser to dime size
Small Pebble	No. 5+	4 - 8 mm	Pea size to pencil eraser
Granule	No. 10 - 5	2 - 4 mm	Rock salt to pea size
Very Coarse Sand	No. 18 - 10	1 - 2 mm	See field gauge card
Coarse Sand	No. 35 -18	0.5 - 1 mm	See field gauge card
Medium Sand	No. 60 - 35	0.25 - 0.5 mm	See field gauge card
Fine Sand	No. 120 - 60	0.125 - 0.25 mm	See field gauge card
Very Fine Sand	No. 230 - 120	0.0625 - 0.125 mm	See field gauge card
Silt and Clay. See SOP for description of fines	Not Applicable	<0.0625 mm	Analyze by pipette or hydrometer

PARTICLE PERCENT COMPOSITION ESTIMATION



GRAPH FOR DETERMINING SIZE OF PARTICLES



FOR COARSE-GRAINED SOILS

Description	Criteria
	Descriptor - Angularity
Angular	Particles have sharp edges and relatively planar sides withunpolished surfaces.
Subangular	Particles are similar to angular but have rounded edges.
Subround	Particles have nearly planar sides but have well-roundedcorners and edges.
Round	Particles have smoothly curved sides and no edges.
Minor Components with Descriptors	
	Sorting Cu= d60/d10
Well Sorted	Near uniform grain-size distribution Cu= 1 to 3.
Poorly Sorted	Wide range of grain size Cu= 4 to 6.
	Moisture
Dry	Absence of moisture, dry to touch, dusty.
Moist	Damp but no visible water.
Wet	Visible free water; soil is usually below the water table. (Saturated)
	Density
Very loose	N-value 1 - 4
Loose	N-value 5 - 10
Medium Dense	N-value 11 - 30
Dense	N-value 31 - 50
Very dense	N-value >50
Color using Munsell	
Geologic Origin (if known)	
Other	
	Cementation
Weak Cementation	Crumbles or breaks with handling or little finger pressure.
Moderate Cementation	Crumbles or breaks with considerable finger pressure.
Strong Cementation	Will not crumble with finger pressure.
	Reaction with Dilute HCl Solution (10%)
No Reaction	No visible reaction.
Weak Reaction	Some reaction, with bubbles forming slowly.
Strong Reaction	Violent reaction, with bubbles forming immediately.

EXAMPLE OF SOIL DESCRIPTION AND PHOTO

10 -15 feet SAND, medium to very coarse, little granules to medium pebbles, subround to subangular, trace silt; poorly sorted, wet, grayish brown (10YR 5/2).



SOIL DESCRIPTION FIELD GUIDE (APRIL, 2022; REV. 3.0)

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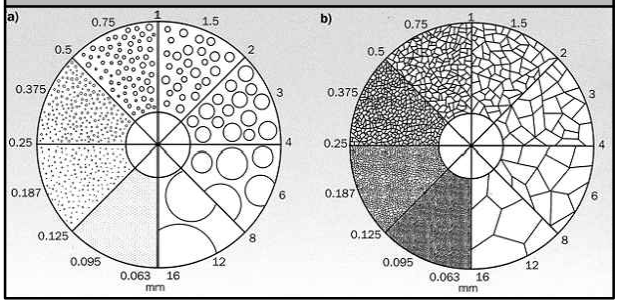
VARIATIONS IN SOIL STRATIGRAPHY

Term	Thickness of Configuration
Parting	0 - to 1/16-inch thickness.
Seam	1/16 - to 1/2-inch thickness.
Layer	1/2 - to 12-inch thickness.
Stratum	> 12-inch thickness.
Pocket	Small erratic deposit, usually less than 1 foot in size.
Varved Clay	Alternating seams or layers of sand, silt, and clay (laminated).
Occasional	≤ 1 foot thick.
Frequent	> 1 foot thick.

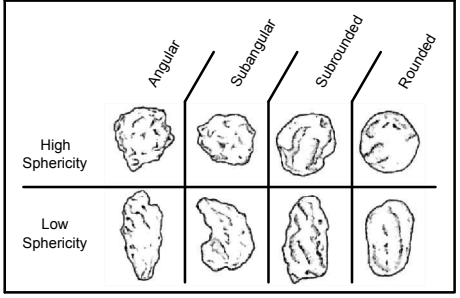
SOIL STRUCTURE DESCRIPTIONS

Term	Description
Homogeneous	Same color and appearance throughout.
Laminated	Alternating layers < 1/4 inch thick.
Stratified	Alternating layers ≥ 1/4 inch thick.
Lensed	Inclusions of small pockets of different materials, such as lenses of sand scattered through a mass of clay; note thickness.
Blocky	Cohesive soil can be broken down into small angular lumps, which resist further breakdown.
Fissured	Breaks along definite planes of fracture with little resistance to fracturing.
Slickensided	Fracture planes appear to be polished or glossy, sometimes striated.

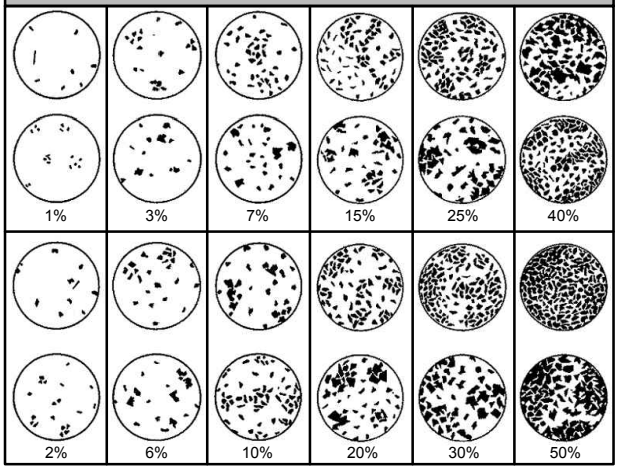
GRAPH FOR DETERMINING SIZE OF PARTICLES



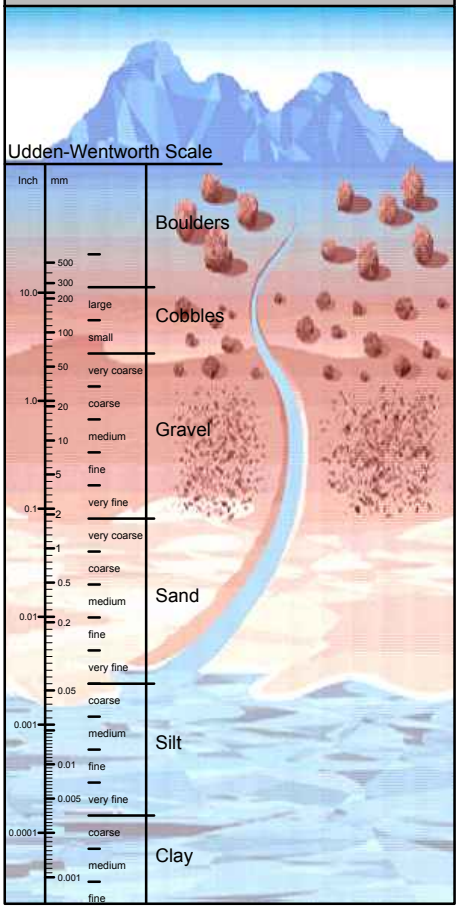
ANGULARITY CHART



PARTICLE PERCENT COMPOSITION ESTIMATION



SORTING



SETTLING TABLE (SILT/CLAY)

Diameter of Particle (mm)	<0.625	<0.031	<0.016	<0.008	<0.004	<0.002	<0.0005
Depth of Withdrawal (cm)	10	10	10	10	5	5	3
Time of Withdrawal	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec
Temperature (Celsius)							
20	00:00:29	00:01:55	00:07:40	00:30:40	00:61:19	04:05:00	37:21:00
21	00:00:28	00:01:52	00:07:29	00:29:58	00:59:50	04:00:00	
22	00:00:27	00:01:50	00:07:18	00:29:13	00:58:22	03:54:00	
23	00:00:27	00:01:47	00:07:08	00:28:34	00:57:05	03:48:00	
24	00:00:26	00:01:45	00:06:58	00:27:52	00:55:41	03:43:00	33:56:00
25	00:00:25	00:01:42	00:06:48	00:27:14	00:54:25	03:38:00	
26	00:00:25	00:01:40	00:06:39	00:26:38	00:53:12	03:33:00	
27	00:00:24	00:01:38	00:06:31	00:26:02	00:52:02	03:28:00	
28	00:00:24	00:01:35	00:06:22	00:25:28	00:50:52	03:24:00	31:00:00
29	00:00:23	00:01:33	00:06:13	00:24:53	00:49:42	03:10:00	
30	00:00:23	00:01:31	00:06:06	00:24:22	00:48:42	03:05:00	

10 inches
9 inches
8 inches
7 inches
6 inches
5 inches
4 inches
3 inches
2 inches
1 inch
1/10in
0 mm
10 mm
20 mm
30 mm
40 mm
50 mm
60 mm
70 mm
80 mm
90 mm
100 mm
110 mm
120 mm
130 mm
140 mm
150 mm
160 mm
170 mm
180 mm
190 mm
200 mm
210 mm
220 mm
230 mm
240 mm
250 mm

Attachment B

Particle Size System Comparison

The purpose of this attachment is to illustrate how the Udden-Wentworth particle sizes and descriptive terms compares to other particle size systems.

When in the field, it is a customary practice to compare current soil descriptions to historical soil boring logs for reference purposes. When reviewing boring logs prepared by others, field staff should first note the particle size system used and recognize these particle size systems may differ. This will avoid confusion when cross referencing between historical and new boring logs and when reviewing existing geologic cross-sections.

For example, a well-sorted sand with grain sizes ranging from 1 to 2 mm should be classified as a very coarse sand by the Udden-Wentworth system. As shown in this attachment, the same particle size would be classified as a medium sand by the United Soil Classification System. The later system has fewer particle size grades and in general, is less descriptive than the Udden-Wentworth system.

PARTICLE SIZE SYSTEM COMPARISON

Grain size distribution in millimeters (mm)

Udden-Wentworth — Remediation Geologists and Engineers

		V. Fine	Fine	Medium	Coarse	V. Coarse	Granule	Pebbles				Cobbles		
								Small	Medium	Large	V. Large	Small	Large	
CLAY	SILT	SAND					GRAVEL							
0.039	0.065	0.125	0.25	0.5	1	2	4	8	16	32	64	128	256	
	1/16	1/8	1/4	1/2										

United Soil Classification System — Geotechnical Engineers

		Fine	Medium	Coarse	Fine	Coarse	
CLAY	SILT	SAND			GRAVEL		COBBLE
	0.074	0.42		2	4.75	19	75	300

U.S. Dept. of Agriculture — Soil Scientists

		V. Fine	Fine	Medium	Coarse	V. Coarse	
CLAY	SILT	SAND					GRAVEL
0.002	0.05	0.10	0.25	0.5	1	2	75

Remediation Hydraulics 2008, page 195): The Udden-Wentworth scale is preferred "...because the geometric progression of grain-size diameter also reflects relationships that are important when considering the erosion and deposition of sediments during the depositional process. The correlation between increasing grain size and degree of sorting and permeability is the most important, as permeability structure is responsible for the mobile and immobile porosity within aquifer systems. "

Attachment C

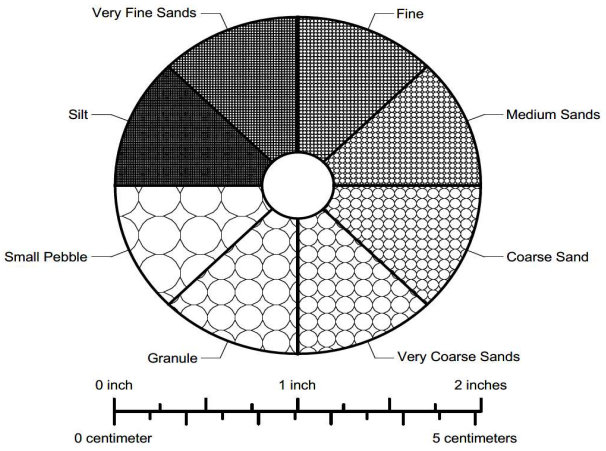
Description of Soil Logging Terms

The purpose of this attachment is to concisely define the soil logging terms used when filling out boring logs. During report preparation, project staff could use this sheet as an index placed in front of the completed boring logs. Also, it can serve as a supplemental reference sheet during field activities.

Description of Logging Terms

Note: Soil descriptions based on Arcadis Technical Guidance and Instructions (TGI) procedures. Key terms defined below.

GRAPH FOR DETERMINING SIZE OF PARTICLES



Udden Wentworth Soil Sizes

Boulder	> 256 mm
Large Cobble	128 to 256 mm
Small Cobble	64 to 128 mm
Very Large Pebble	32 to 64 mm
Large Pebble	16 to 32 mm
Medium Pebble	8 to 16 mm
Small Pebble	4 to 8 mm
Granule	2 to 4 mm
Very Coarse Sand	1 to 2 mm
Coarse Sand	0.5 to 1 mm
Medium Sand	0.25 to 0.5 mm
Fine Sand	0.125 to 0.25 mm
Very Fine Sand	0.062 to 0.12 mm
Silt/Clay	<0.065 mm

Primary Texture *(e.g. CLAY, SILT, SAND, GRANULE, PEAT, MUCK, FILL, etc.)*
List particle size with the highest percentage per sample interval (e.g. SAND)
Always CAPITALIZE the primary texture
Follow primary texture with a comma followed by grain-size descriptors, etc.

Minor Texture

And	(36 to 50%)
Some	(21 to 35%)
Little	(10 to 20%)
Trace	(>10%)

Angularity

Angular	Sharp edges
Sub-Angular	Rounded edges
Sub-Rounded	Well-rounded
Rounded	Smooth curved edges

Sand Density (Blow Counts/ft)

Very Loose	0-4
Loose	5-10
Medium Dense	11-30
Dense	31-50
Very Dense	<50

Silt/Clay Consistency (Blow Counts/ft)

Very Soft	0-2,	thumb easily penetrates several inches
Soft	3-4,	thumb easily penetrates one inch
Medium Stiff	5-8,	thumb indents 0.5 in. with much effort
Stiff	9-15,	thumb indents 0.25 in. with great effort
Very Stiff	16-30,	thumbnail is readily intended

Sorting

Well Sorted	1 to 3 Particle Sizes
Poorly Sorted	4+ Particle Sizes

Moisture Content

Dry	Dry to touch
Moist	No visible water
Wet	Visible free water

Plasticity (for silts and clays)

Non-Plastic	3 mm thread can not be rolled
Low Plasticity	3 mm thread can barely be rolled
Medium Plasticity	3 mm thread can easily and quickly rolled, but not rerolled
High Plasticity	3 mm thread can be rolled slowly, but can be rerolled

Dilatancy (for silts and silt-sand mixtures)

None	No visible change in the specimen
Slow	Water appears slowly during shaking / disappears slowly or not at all upon squeezing
Rapid	Water appears quickly during shaking / disappears quickly upon squeezing

Example Description

10 -15 feet SAND, medium to very coarse, little granules to medium pebbles, subround to subangular, trace silt; poorly sorted, wet, grayish brown (10YR5/2).

Attachment D

Blank Boring Log

The purpose of this attachment is to present a blank field form for use during soil logging. A digital version (Microsoft Excel) of this field form is available from the authors (upon request). If project specific modifications to this boring log template are warranted, please contact the Site Investigation Community of Practice leader for further assistance.

BORING LOG

ARCADIS

Boring ID:		**Project Name:**		**Page:**	/
Permit ID:		**Date Started:**		**Ground Elevation:**	
Site Address:		**Date Completed:**		**Vertical Datum:**	
City, State:		**Total Depth:**		**Northing:**	
Drilling Co:		**Depth to Water:**		**Easting:**	
Driller:		**Hole Diameter:**		**Horizontal Datum:**	
Drilling Method:		**Core Device:**		**Prepared by:**	
Boring Status:		**Drilling Fluid:**		**Reviewed by:**	

Drilling Information				Graphical Log for Primary Texture									Soil Description (Udden-Wentworth System)	Field Notes
				Fines		Sand					Gravel			
Drilling Depth (ft bgs)	Core Interval (ft)	Core Recovery (inches)	Vapor Reading (ppm)	clay	silt	very fine	fine	medium	coarse	very coarse	granule	pebble	cobble	boulder	Depth Interval (ft), PRIMARY TEXTURE, Principal and Minor Components with Descriptors (% modifiers and grain size fraction, angularity for coarse sand and larger, consistency/density, plasticity for silt and clay, dilatancy for silt/silt-sand); Sorting, Moisture Content, Color. NOTES: *Texture Modifiers: Trace (<10%), Little (10 to 20%), Some (21 to 35%), And (36 to 50%)*	Driller's Observations, Geologic Formation, Field Screening Results, Sample Interval etc.

BORING LOG

Boring ID: _____ **Project Name:** _____ **Page:** _____ / _____

Drilling Information				Graphical Log for Primary Texture										Soil Description (Udden-Wentworth System)	Field Notes
				Fines		Sand					Gravel				
Drilling Depth (ft bgs)	Core Interval (ft)	Core Recovery (inches)	Vapor Reading (ppm)	clay	silt	very fine	fine	medium	coarse	very coarse	granule	pebble	cobble	boulder	Depth Interval (ft), PRIMARY TEXTURE, Principal and Minor Components with Descriptors (% modifiers and grain size fraction, angularity for coarse sand and larger, consistency/density, plasticity for silt and clay, dilatancy for silt/silt-sand); Sorting, Moisture Content, Color. NOTES: *Texture Modifiers: Trace (<10%), Little (10 to 20%), Some (21 to 35%), And (36 to 50%)*	Driller's Observations, Geologic Formation, Field Screening Results, Sample Interval etc.

Attachment E

Completed Boring Log

The purpose of this attachment is to provide an example of a completed boring log for reference purposes to field staff. The example provided is for a soil boring completed outside the waste mass of a closed municipal landfill near Baltimore, Maryland. The objective of the drilling program was to determine the depth to groundwater to determine the appropriate depth interval to install a soil gas monitoring well and groundwater monitoring well across the first water-bearing zone. The site geology consists of unconsolidated sediments of the Mid-Atlantic Coastal Plain, specifically the Upper Patapsco formation. These sediments were deposited in a moderate gradient fluvial environment during the Cretaceous period. The landfill was constructed into a regional clay confining unit.

BORING LOG

ARCADIS

Boring ID:	MW-08	**Project Name:**	Acme Landfill	**Page:**	1 / 1
Permit ID:	MD-PG-100	**Date Started:**	7/18/2018	**Ground Elevation:**	50.5 ft
Site Address:	100 Landfill Road	**Date Completed:**	7/18/2018	**Vertical Datum:**	NAVD 88, feet
City, State:	Baltimore, Maryland	**Total Depth:**	35 ft below ground	**Northing:**	123456.79
Drilling Co:	Earth Matters	**Depth to Water:**	19 ft below ground	**Easting:**	123456.79
Driller:	Rod E. Piper	**Hole Diameter:**	2-inch	**Horizontal Datum:**	NAD 83 feet, MD State
Drilling Method:	Direct-push/hollow-stem	**Core Device:**	5-foot macrocore sampler	**Prepared by:**	Sandy Pebbles
Boring Status:	completed as well	**Drilling Fluid:**	none	**Reviewed by:**	Clay Brown

Drilling Information				Graphical Log for Primary Texture										Soil Description (Udden-Wentworth System)	Field Notes
				Fines		Sand					Gravel				
Drilling Depth (ft bgs)	Core Interval (ft)	Core Recovery (inches)	VOC Vapor Reading (ppm)	clay	silt	very fine	fine	medium	coarse	very coarse	granule	pebble	cobble	boulder	Depth Interval (ft), PRIMARY TEXTURE, Principal and Minor Components with Descriptors (% modifiers and grain size fraction, angularity for coarse sand and larger, consistency/density, plasticity for silt and clay, dilatancy for silt/silt-sand); Sorting, Moisture Content, Color. NOTES: *Texture Modifiers: Trace (<10%), Little (10 to 20%), Some (21 to 35%), And (36 to 50%)*	Driller's Observations, Geologic Formation, Field Screening Results, Sample Interval etc.
0 to 1			< 1												0-0.5 ft, topsoil with organics	Grass covered area
1 to 2			< 1				X								0.5-5 ft, SAND, fine, trace silt, trace pebble, round; poorly sorted, moist, yellowish brown (7.5 YR 5/8). NOTE: some cementation, does not react with HCl	continuous macro-core logging
2 to 3	0-5	43.2/60	< 1				X									
3 to 4			< 1				X									cemented sand @3.6-4 ft
4 to 5			< 1				X									
5 to 6			< 1				X	X	X						5-10 ft, SAND, fine to coarse, round to subround; well sorted, moist, light to strong brown (7.5 YR 6/4 to 7.5 YR 5/6).	
6 to 7			< 1				X	X	X							
7 to 8	5-10	40.8/60	< 1				X	X	X							
8 to 9			< 1				X	X	X							
9 to 10			< 1				X	X	X							
10 to 11			< 1				X	X	X						10-12.5 ft, same as above with trace silt	
11 to 12			< 1				X	X	X							
12 to 13	10-15	36/60	< 1				X	X	X							
13 to 14			< 1				X	X	X						12.5 to 15 ft, same as above, color change to pink (7.5 YR 7/3) and reddish yellow (7.5YR 6/8)	
14 to 15			< 1				X	X	X							
15 to 16			< 1						X	X					15-18.9 ft, SAND, coarse to very coarse, round to subround; well sorted, moist, strong brown (7.5YR 5/6) to reddish yellow (7.5YR 6/6)	
16 to 17			< 1						X	X						
17 to 18	15-20	55.2/60	< 1						X	X						
18 to 19			< 1		X	X	X								18.9-22.7 ft, SAND, very fine to fine, and SILT, coarse to very coarse, poorly sorted, wet, light gray (7.5YR 7/1)	water table encountered @ 18.9 ft
19 to 20			< 1		X	X	X									
20 to 21			< 1		X	X	X									
21 to 22			< 1		X	X	X									
21 to 23	20-25	36/60	< 1		X	X	X									
23 to 24			< 1	X	X										22.7-25 ft, CLAY and SILT, high plasticity, soft to stiff at 25 ft, dry to moist, light gray (2/5YR 7/1) w/ red mottling (2.5YR 4/6)	Middle Patapsco Confining Unit
24 to 25			< 1	X	X											
25 to 26			< 1	X	X										25-31.1 ft, CLAY and SILT, high plasticity, stiff; dry to moist, light gray (2/5YR 7/1) with red mottling (2.5YR 4/6)	
26 to 27			< 1	X	X											
27 to 28	25-30	30/60	< 1	X	X											
28 to 29			< 1	X	X											
29 to 30			< 1	X	X											
30 to 31			< 1	X	X											
31 to 32			< 1		X											
32 to 33	30-35 ft	60/60	< 1		X											
33 to 34			< 1		X										31.1-35 ft, SILT, low plasticity, high dilatancy; wet, gray (7.5YR 7/1)	End of direct-push boring @ 35 ft
34 to 35			< 1		X											

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TGI – Soil Drilling and Sample Collection

Rev: #3

Rev Date: April 5, 2023

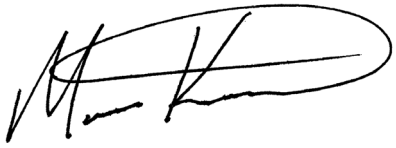
Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
	0	October 11, 2018	All	Updated and re-written as a TGI	Marc Killingstad
	1	May 12, 2020	None	Review – no changes necessary	Marc Killingstad
	2	April 8, 2022	All	Updated to new format and minor content (e.g., PFAS)	Chris Shepherd/Marc Killingstad
	3	April 5, 2023	All	Annual review completed by Marc Killingstad. Updated document revision number, document date; version control and signature page.	Marc Killingstad

Approval Signatures

Prepared by: 4/3/2023

Chris Shepherd (Preparer) Date

Reviewed by:  4/3/2023

Marc Killingstad (Subject Matter Expert) Date

1 Introduction

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM. All deviations or omissions should be documented.

2 Intended Use and Responsibilities

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3 Scope and Application

This Technical Guidance Instruction (TGI) describes general drilling procedures and the methods to be used to field screen and collect soil samples for laboratory analysis in unconsolidated or weakly consolidated sediments. For soil description procedures, please refer to the *TGI - Soil Description*. For monitoring well installation in granular aquifers, please refer to the *TGI - Monitoring Well Installation*. For per- and polyfluoroalkyl substances (PFASs) drilling and soil sampling procedures, please refer to: *TGI – PFAS-Specific Drilling and Monitoring Well Installation, TGI – Per- and Polyfluoroalkyl Substances (PFAS) Field Sampling Guide*, and *TGI – Equipment and Reagent Blank Sample Collection for PFAS Analysis*.

Overburden (unconsolidated sediments) drilling is commonly performed using the hollow-stem auger drilling method. Other drilling methods suitable for overburden drilling, which are sometimes necessary due to site-specific geologic conditions, include: direct-push, drive-and-wash, spun casing, rotasonic, dual-rotary (Barber Rig), and fluid/mud rotary with core barrel or roller bit. Direct-push techniques (e.g., Geoprobe or cone penetrometer) and hand tools may also be used. Drilling within consolidated materials such as fractured rock is commonly performed using water-rotary (coring or tri-cone roller bit), air rotary or rotasonic methods. For guidance when drilling in consolidated materials (i.e., bedrock), please refer to *the TGI – Bedrock Core Collection and Description.*

The drilling method to be used at a given site will be selected based on site-specific consideration of anticipated drilling depths, targeted chemicals, site or regional geologic knowledge, types of sampling to be conducted, required sample quality and volume, and cost.

Field screening of soil samples is commonly performed using a photoionization detector (PID) and/or a flame ionization detector (FID). These instruments are used to measure relative concentrations of volatile organic compounds (VOCs) for the selection of samples for further laboratory or field analysis. Field screening for dense non-aqueous phase liquids (DNAPL) may be performed using hydrophobic dye (Oil Red O or Sudan IV), which is pertinent at chlorinated solvent sites.

Collection of soil samples for laboratory analysis may be performed using a variety of techniques including grab samples, undisturbed cores, and composite or homogenized samples. Samples may require homogenization across a given depth interval, or several discrete grabs (usually five) may be combined into a composite sample. Samples for VOC analysis will not be homogenized or composited and are collected as discrete grab samples.

No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools). Some lubricants (e.g., vegetable oil-based lubricants) may be acceptable, if the constituents won't interfere with the analyses.

4 Personnel Qualifications

Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or state/federal regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid,

cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, Arcadis field personnel will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP), Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis field personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Personnel responsible for overseeing drilling operations will have at least 16 hours of prior training overseeing drilling activities with an experienced geologist, environmental scientist, or engineer with at least 2 years of prior experience.

Arcadis personnel directing, supervising, or leading soil sampling activities will have a minimum of 1 year of previous environmental soil sampling experience. Field employees with less than 6 months of experience will be accompanied by a supervisor (as described above) to ensure that proper sample collection techniques are employed.

Additionally, the Arcadis field team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

5 Equipment List

The following materials will be available, as required, during soil boring drilling, field screening, andsampling activities:

- Site-specific HASP and health and safety documents identified in the HASP
- FIP/work plan that includes site map with proposed boring locations, fieldsampling plan (with corresponding depths, sample analyses, sample volume required, and sample holding time), and previous boring logs (as available)
- Appropriate personal protective equipment (PPE), as specified in the HASP
- Including but not limited to disposable chemical resistant gloves and Level D PPE
- Traffic cones, delineators, and caution tape as appropriate for securing the work area as specified in the Traffic Safety Plan (TSP)
- Photoionization detector (PID), flame ionization detector (FID) or other air/soil screening equipment, asneeded, in accordance with the HASP or workplan
- Sampling equipment:
- Drilling equipment required by *ASTM D1586*, when performing split-spoon sampling including clean sample sleeves
- Disposable plastic liners, when drilling with direct-push equipment
- Stainless steel hand auger and stainless-steel spade if using manual methods
- Appropriate soil sampling equipment (e.g., stainless steel spatulas/spoons/bowls, knife)
- Sealable plastic bags (e.g., Ziploc®)

- Air-tight sample containers and 8-oz. glass Mason jars or driller's jars
- Aluminum foil
- Appropriate sample blanks (trip blank supplied by the laboratory), as specified in the FSP
- Soil sample containers and labels (supplied by the laboratory) appropriate for the analytical method(s) with preservative, as needed (parameter-specific)
- Sample labels
- Indelible ink pens
- Engineer's ruler or survey rod
- Plastic sheeting (e.g., Weatherall Visqueen)
- Appropriate transport containers (coolers) with ice and appropriate labeling, packing, and shipping materials
- Decontamination equipment (buckets, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels) in accordance with the *TGI for Groundwater and Soil Sampling Equipment Decontamination*
- Forms/notes:
    - Tablet with digital forms, etc., if appropriate
    - Appropriate soil boring log (**Attachment 1**)
    - Chain-of-custody forms
    - Field notebook
    - Digital camera (or smart phone with camera)
- Drums or other containers appropriate for soil and decontamination water, as specified by the site investigation-derived waste (IDW) management plan, and appropriate drum labels

6 Cautions

Prior to beginning field work, underground utilities in the vicinity of the drilling areas will be delineated by the drilling contractor or an independent underground utility locator service in accordance with the work plan, client requirements, and Arcadis guidance. See appropriate guidance forproper utility clearance protocol. Work will be performed in accordance with the Arcadis *Utility Location and Clearance Health and Safety Standard* and the *Utilities and Structures Checklist* will be completed before beginning any intrusive work.

Prior to beginning field work, the project technical team will ensure that all field logistics (e.g., access issues, health and safety issues, communication network, schedules, etc.) and task objectives are clearly understood by all team members. An internal call with the project technical team to review the FIP/work plan scope and objectives is strongly recommended prior to mobilization to ensure that the field work will be effectively and efficiently executed.

Some regulatory agencies have specific requirements regarding borehole abandonment and grout mixtures. Determine whether the oversight agency has any such requirements prior to finalizing the

drilling plan.

If DNAPL is known or expected to exist at the site, refer to the project specific documents (e.g., DNAPL Contingency Plan) for additional details regarding drilling to reduce the potential for inadvertent DNAPL remobilization.

Similarly, if light non-aqueous phase liquid (LNAPL) is known or expected to be present as “perched” layers above the water table, refer to the DNAPL Contingency Plan. Follow the general provisions and concepts in the DNAPL contingency plan during drilling above the water table at known or expected LNAPL sites.

Avoid using drilling fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions. Water used for drilling, decontamination of drilling/sampling equipment, or grouting boreholes uponcompletion will be of a quality acceptable for project objectives. Testing of water supply will be considered.

Specifications of materials used for backfilling the borehole will be obtained, reviewed and approved to meet project quality objectives. Bentonite is not recommended where DNAPL is likely to be present or in groundwater with high salinity. In these situations, neat cement grout is preferred.

Store and/or stage empty and full sample containers and coolers out of direct sunlight. Sample container threads should be wiped down with a clean, nonabrasive material (e.g., paper towels) to better ensure the sample container is properly sealed. Be careful not to over-tighten lids with Teflon® liners or septa. Over-tightening can impair the integrity of the seal and cancause the glass to shatter and create a risk for hand injuries.

NOTE: *Field logs and some forms are considered to be legal documents. All field logs and forms will therefore be filled out in indelible ink. Do not use permanent marker or felt-tipped pens for labels on sample container or sample coolers. Permanent markers could introduce volatile constituents into the samples.*

NOTE: *An Arcadis employee that is appropriately trained at the correct level of internal hazardous materials/DOT (Department of Transportation) shipping must complete an Arcadis shipping determination to address applicable DOT and IATA (International Air Transport Association) shipping requirements. Review the applicable Arcadis procedures and guidance instructions for sample packaging and labeling. Prior to using air transportation, confirm air shipment is acceptable under DOT and IATA regulations.*

7 Health and Safety Considerations

The HASP will be followed, as appropriate, to ensure the safety of field personnel. Review all site-specific and procedural hazards as they are provided in the HASP, and review Job SafetyAnalysis (JSA) documents in the field each day prior to beginning work.

Prior to drilling, utility clearance must be performed (see Section 5). Appropriate personal protective equipment (PPE) will be worn at all times in line with the task and thesite-specific HASP.

Working outside at sites with suspected contamination may expose field personnel to hazardous materials such as contaminated groundwater or NAPL (e.g., oil). Other potential hazards include biological hazards (e.g., stinging insects, ticks in long grass/weeds, etc.), and potentially the use of sharp cutting tools (scissors, knife). Only use non-toxic peppermint oil spray for stinging insect nests. Review client-specific health and safety requirements, which may preclude the useof fixed/folding-blade knives

and use appropriate hand protection.

If thunder or lightning is present, discontinue drilling and sampling until 30 minutes have passed after the last occurrence of thunder or lightning.

8 Procedure

The procedures for drilling and the methods to be used to field screen and collect soil samples for laboratory analysis are presented below:

8.1 Drilling Procedures

8.1.1 Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods

1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area.
    a. Verify utilities were cleared (see Section 5) and use soft dig technique to clear borehole, if applicable
    b. Clean sampling equipment in accordance with the FIP/work plan prior to drilling
2. Advance boring to target depth:
    a. Collect soil samples at appropriate interval as specified in the FIP/work plan (or equivalent) using the appropriate tooling (e.g., split-barrel sampler) and sample containers
        i. Split-barrel or drive-ahead samples are obtained during drilling
        ii. A common sampling method that produces high-quality soil samples with relatively littlesoil disturbance is described in *ASTM D1586 – Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils* (ASTM D1586).
    b. Always change disposable gloves before handling the sampling equipment
    c. Collect, document, and store samples for laboratory analysis as specified in the FIP/work plan (or equivalent; see below for additional details on sample collection procedures)
    d. Field screen samples as specified in the FIP/work plan (or equivalent; see below for additional details on field screening procedures)
    e. Rotasonic drilling produces soil cores that, for the most part, are relatively undisturbed, but note that when drilling in consolidated or finer-grained sediment the vibratory action during core barrel advancement may create secondary fractures or breaks. The core is retrieved by vibrating the soil/rock into a separate core bag, typically in 5-foot or 10-foot increments. The soil cores may consolidate or expand during retrieval, depending on soils, etc.
    f. Dual-rotary removes cuttings by compressed air or water/mud and allow only a

generalassessment of geology unless separate coring tools and techniques are used

g. Decontaminate equipment between samples in accordance with the FIP/work plan (or equivalent)

3. Describe each soil sample as outlined in the appropriate project records (refer to the description procedures outlined in the *TGI - Soil Description*)

   a. Record descriptions on the soil boring log (**Attachment 1**) and/or field notebook

   b. When possible, photo document the samples (e.g., soil cores, split-barrels)

   c. During soil boring advancement, document all drilling events in field notebook, including blow counts (i.e., the number of blows from a soil sampling drive weight [140 pounds] required to drive the split-barrel sampler in 6-inch increments) and work stoppages

   d. Blow counts will not be available if rotasonic, dual-rotary, or direct-push methods are used; however, if standard penetration testing is required during rotasonic drilling, an automatic drop hammer may be used in conjunction with the method to switch from core barrel advancement to standard penetration testing

   e. If soils are screened with a PID/FID or another instrument, document the measurement in accordance with the work plan

4. The drilling contractor will be responsible for obtaining accurate and representative samples, informing the supervising Arcadis geologist of changes in drilling pressure, drilling penetration rates, and keeping a separategeneral log of soils encountered, including blow counts

   a. The term “samples” means soil materials from particular depth intervals, whether or not portions of these materials are submitted for laboratory analyses

   b. Records will also be kept of occurrences of premature refusal due to boulders, construction materials that may have been used as fill, etc.

   c. Where a boring cannot be advanced to the desired depth, the boring will be abandoned, and an additional boring will be advanced at an adjacent location to obtain the required sample in accordance with the work plan

   d. Where it is desirable to avoid leaving vertical connections between depth intervals (e.g., if DNAPL or perched LNAPL are known or expected to exist at the site), the borehole will be sealed using cement and/or bentonite (see **Section 5** above)

   e. Multiple refusals may lead to a decision by the supervising geologist to abandon that sampling location

8.1.2 Direct-Push Method

The direct-push drilling method may also be used to complete soil borings. Examples of this technique include Geoprobe®, Diedrich Environmental Soil Probe (ESP) System, or AMS PowerProbe.

Environmental probe systems typically use a hydraulically operated percussion hammer.

Depending on the equipment used, the hammer delivers 140- to 350-foot pounds of energy with each blow. The hammer provides the force needed to penetrate very stiff to medium dense soil formations. The hammer simultaneously advances an outer steel casing that contains a dual tube liner for samplingsoil

(dual tube sampling system).

The outside diameter (OD) of the outer casing ranges from 2.25 to 6 inches and the OD of the inner sampling tube diameter ranges from 1.4 to 4.5 inches. The outer casing isolates overlying soil and permits the unit to continue to probe at depth. The dual tube sampling system provides a borehole that may be tremie-grouted from the bottom up. Alternatively, a single rod system may be used that does notprovide a cased boring and which limits tremie-grouting from the bottom up.

Direct-push drilling can generally achieve target depths 100 feet or less depending on the site geology. The known or expected site conditions (e.g., presence of NAPL) will be evaluated when selecting the typeof direct-push sampling system to be employed.

1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area
    a. Verify utilities were cleared (see Section 5) and use soft dig technique to clear borehole, if applicable
    b. Clean sampling equipment in accordance with the FIP/work plan prior to drilling
2. Advance soil boring to target depth.
    a. Collect soil samples at appropriate interval as specified in the FIP/work plan (or equivalent) using clean/disposable sampling equipment (plastic liners)
    b. Always change disposable gloves before handling the sampling equipment
    c. Collect, document, and store samples for laboratory analysis as specified in theFIP/work plan (or equivalent; see below for additional details on sample collection procedures)
    d. Field screen samples as specified in the FIP/work plan (or equivalent; see below for additional details on field screening procedures)
3. Decontaminate equipment between samples in accordance with the FIP/work plan (or equivalent)
4. Describe samples in accordance with the procedures outlined in **Step 3** under ***Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods*** above (refer to the description procedures outlined in the *TGI - Soil Description*)

8.1.3 Manual Methods

Manual methods may also be used to complete shallow soil borings. Examples of this technique include using a spade, spoon, scoop, hand auger, or slide hammer. Manual methods are typically used to collect surface soil samples (0 to 6 inches) or to complete soil borings/collect soil samples from a depth of 5 feet or less.

1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area
2. Clear the ground surface of brush, root mat, grass, leaves, or other debris
3. Use a spade, spoon, scoop, hand auger, or slide hammer to collect a sample of the required depth interval
4. Use an engineer's ruler or survey rod to verify that the sample is collected to the correct depth and

record the top and bottom depths from the ground surface

5. To collect samples below the surface interval, remove the surface interval first; then collect the deeper interval
    a. To prevent the hole from collapsing, it may be necessary to remove a wider section from the surface or use cut polyvinyl chloride (PVC) pipe to maintain the opening
    b. Collect soil samples at appropriate interval as specified in the FIP/work plan (or equivalent) and transfer to the appropriate, laboratory-supplied container
    c. Collect, document, and store samples for laboratory analysis as specified in the FIP/work plan (or equivalent; see below for additional details on sample collection procedures)
    d. Field screen samples as specified in the FIP/work plan (or equivalent; see below for additional details on field screening procedures)
6. Decontaminate equipment between samples in accordance with the FIP/work plan (orequivalent)
7. Describe samples in accordance with the procedures outlined in **Step 3** under ***Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods*** above (refer to the description procedures outlined in the *TGI - Soil Description*)

8.2 Field Screening Procedures

8.2.1 PID and FID Screening

Soils are typically field screened with a PID or FID for a relative measure of the total VOCs at sites where VOCs are known or suspected to exist. PIDs and FIDs require calibration in accordance with the work plan(s) and manufacturer's specifications and PIDs should be calibrated based on the target chemicals. The PID employs an ultraviolet lamp to measure VOCs and the ionizationenergy (IE) of the site constituents need to be considered when selecting the type of lamp (e.g., 10.6 eV, 11.7 eV) that will be used. In general, any compound with an IE lower than that of the lamp photons canbe measured. The FID has a wide linear range and responds to almost all VOCs.

Field screening is performed using one (or both) of the following two methods:

1. Upon opening the sampler, the soil is split open and the PID or FID probe is placed in the opening and covered with a clean, gloved hand. Such readings will be obtained at several locations along the lengthof the sample.
2. A portion of the collected soil is placed in a jar, which is covered with aluminum foil, sealed, and allowed to warm to room temperature (see below). After warming, the cover is removed, the foil is pierced withthe PID or FID probe, and a reading is obtained.

Prior to usage, the PID or FID must be calibrated according to the manufacturer's specifications at a minimum frequencyof once per day prior to collecting PID or FID readings. The PID will be calibrated to a benzene-related compound (isobutylene) or other appropriate gas, while the FID will be calibrated to methane. The time, date, and calibration procedure must be clearly documented in the field notebook and/or the calibration form.

If at any time the PID or FID results appear erratic or inconsistent with field observations, then the instrument will be recalibrated.

If calibration is difficult to achieve, then the PID's lamp will be checked for dirt or moisture and cleaned, or technical assistance will be required. Maintenance and calibration records will be kept as part of the field quality assurance program.

Initial PID readings will be recorded on the soil boring log (**Attachment 1**) and/or in the field notes. The soil sample will be separated from the slough material (if any) by using disposable gloves and a pre-cleaned stainless-steel spoon or tool.

For the second method, a representative portion of the sample will be placed in a pre-cleaned air-tight container (as quickly as possible to avoid loss of VOCs), filling the container half full to allow for the accumulation of vapors above the soil. An aluminum foil seal will be placed between the glass and cap and the cap will be screwed on tightly. Unless the screening will be performed immediately after the sample is placed in the container, the sample containers will be stored in a cooler chilled to approximately4ºC until screening can be performed.

The headspace of the container will be measured using a PID or FID as follows:

1. Samples will be taken to a warm workspace and allowed to equilibrate to room temperature for atleast one hour.
2. Prior to measuring the soil vapor headspace concentration, the container will be shaken.
3. The headspace of the sample will then be measured directly from the container by piercingthe aluminum foil seal with the probe of the PID or FID and measuring the relative concentration of VOCs in the headspace of the soil sample. The initial (peak) reading must be recorded.

8.2.2 NAPL Screening

To screen for the potential presence of non-aqueous phase liquid (NAPL) in soil, drilling procedures must allow for high-quality porous media samples to be taken. Split-barrel samplers or direct-push samplers will be collected continuously ahead of the auger, drill casing/rods, or probe rods. Upon opening each split-barrel sampler or direct-push plastic liner sleeve, the soil will immediately be evaluated for the presence of visible NAPL and odors. If suspected NAPL is immediately visible in the sample, its depth will benoted.

Additionally, the soil will be screened for the presence of organic vapors using a PID or FID, in accordance with the work plan, if applicable. During screening, the soil will be split open using a clean spatula or knife and the PID or FID probe will be placedin the opening and covered with a clean, gloved hand (**Method 1** above). Such readings will be obtained along the entire length of the sample. Alternatively, **Method 2** for PID/FID screening (outlined above) may also be performed. If the PID or FID examination reveals the presence of organic vapors above 100 parts permillion (ppm), the sample will undergo further detailed evaluation for visible NAPL.

The assessment for NAPL will include the following tests/observations:

- Evaluation for Visible NAPL Sheen or Free-Phase NAPL in Soil Sampler
    - NAPL sheen will be a colorful iridescent appearance on the soil sample
    - NAPL may also appear as droplets or continuous accumulations of liquid with a color typically ranging from yellow to brown to black, depending on the type of NAPL
    - Creosote DNAPL (associated with wood-treating sites) and coal tar DNAPL (associated with manufactured gas plant [MGP] sites) are typically black and have a characteristic, pungent odor
    - Pure chlorinated solvents may be colorless in the absence of hydrophobic dye. Solvents mixed

with oils may appear brown

- Particular care will be taken to fully describe any sheens observed, staining, discoloration, droplets (blebs), or NAPL saturation

- Soil-Water Pan Test
  - A portion of the selected soil interval with the highest PID or FID reading above 100 ppm will be placed in a disposable polyethylene dish along with a small volume of potable or distilled water
  - The dish will be gently tilted back and forth to mix the soil and water, and the surface of the water will be viewed in natural light to observe the development of a sheen, if any
  - A small quantity of Oil Red O or Sudan IV hydrophobic dye powder should be added in accordance with the work plan, and the soil and dye will be manually mixed for approximately 30 to 60 seconds and smeared in the dish tocreate a paste-like consistency
  - A positive test result will be indicated by a sheen on the surface of the water and/or a bright red color imparted to the soil following mixing with dye
- Soil-Water Shake Test
  - A small quantity of soil (up to 15 cc) will be placed in a clear, colorless, jar containing an equal volume of potable or distilled water (40-mL vials are well suited to this purpose, but not required)
  - After the soil settles into the water, the surface of the water will be evaluated for a visible sheen under natural light
  - The jar will be closed and gently shaken for approximately 10 to 20 seconds
  - Again, the surface of the water will be evaluated for a visible sheen or a temporary layer of foam
  - A small quantity (approximately 0.5 to 1 cc) of Oil Red O or Sudan IV powder will be placed in the jar in accordance with the work plan
  - The sheen layer, if present, will be evaluated for a reaction to the dye (change to bright red color)
  - The jar will be closed and gently shaken for approximately 10 to 20 seconds
  - The contents in the closed jar will be examined under natural light for visible bright red dyed liquid inside the jar

- A positive test result will be indicated by the presence of a visible sheen or foam on the surface ofwater, a reaction between the dye and the sheen layer upon first addition of the dye powder, a bright red coating on the inside of the vial (particularly above the water line), or red-dyed droplets within the soil

NOTE: *If NAPL is obviously present upon opening the soil sampler or evaluating the soil sample within the split-spoon sampler or direct-push liner sleeve, it is not necessary to perform a soil-water pan test or soil-water shake test. In addition, it is not necessary to perform both a soil-water pan test and a soil-water shake test; either test method is acceptable. The pan test may be preferred in some circumstances because the presence of a sheen may be easier to see on a wider surface. Further, these tests will only be performed if specified in the work plan(s).*

NOTE: *When using hydrophobic dye in the tests above, color will be assessed outdoors under natural light during the period between sunrise and sunset, regardless of the degree of cloud cover. The hydrophobic dye Safety Data Sheets (SDS) will be incorporated into the HASP and reviewed prior to use and the dyes will be carefully handled and disposed in accordance with regulations, if applicable*.

8.3 Soil Sample Collection for Laboratory Procedures

If not specifically identified in the FIP/work plan, soil samples will be selected for laboratory analysis based on:

1. Their position in relation to identified source areas
2. The visual presence of source residues (e.g., NAPL or staining)
3. The relative levels of total VOCs based on field screening measurements
4. The judgment of the field coordinator
5. Moisture content or relative position with regard to apparent groundwater table/saturation

Samples designated for laboratory analysis will be placed in the appropriate containers.

Sample containers for VOC analysis will be filled first immediately following soil core retrieval to reduce loss of VOCs.

If samples will be collected for other analyses, a sufficient amount of the remaining soil willthen be homogenized as described below and sample containers will be filled for other parameters.

VOC samples will be collected as discrete samples using a small diameter core sampler (e.g., En Core® Sampler, Terra Core™ Sampler).

The En Core® Sampler is a disposable volumetric sampling device that collects, stores and delivers soil samples without in-field chemical preservation. The En Core® Sampler requires the use of a reusable T-handle.

The Terra Core™ Sampler is a one-time use transfer tool, designed to collect soil samples and transfer them to the appropriate containers for in-field chemical preservation (e.g., methanol).

The small diameter core samplers will be used according to the manufacturer's instructions (e.g., En Novative Technologies). Some regulatory agencies have specific requirements regarding VOC sample

collection. Determine whether the oversight agency has specific requirements prior to commencing sampling and collect samples at appropriate interval as specified in the FIP/work plan (or equivalent). Samples may require homogenization across a given depth interval, or several discrete grabs (usually five) may be combined into a composite sample.

NOTE: *Samples for VOC and PFAS analysis will NOT be homogenized or composited and will be collected asdiscrete samples as described above*.

The procedure for mixing samples is provided below.

1. Mix the materials in a stainless steel (or appropriate non-reactive material) bowl using a stainless-steel spoon (or disposable equivalents)

a. When dealing with large sample quantities, use disposable plastic sheeting and a shovelor trowel

b. NOTE: *When preparing samples for metals analyses, do not use disposable aluminum(or metal tools or trays other than stainless steel), as it may influence the analytical results*

2. Flatten the pile by pressing the top without further mixing

3. Divide the circular pile by into four equal quarters by dividing out two diameters at right angles

4. Mix each quarter individually using appropriate non-reactive bowls, spoons and/or sheeting

5. Mix two quarters (as described above) to form halves, then mix the two halves to form a composite orhomogenized sample

6. Place composite or homogenized sample into specified containers

7. Remaining material will be disposed of in accordance with project requirements and applicableregulations

8. Sample containers will be labeled with sample identification number, date, and time of collection andplaced on ice in a cooler (target 4° Celsius)

9. Samples selected for laboratory analysis will be documented (chain-of-custody forms), handled, packed, and shipped in accordance with the procedures outlined in the FIP/work plan (or equivalent).

8.4 Soil Boring Abandonment

All soil borings need to be abandoned in accordance with ***TGI for Monitoring Well and Soil Boring Decommissioning***. See Attachment E of the TGI for specifics.

9 Waste Management

Investigative-Derived Waste (IDW) generated during drilling activities, including soil and excess drilling fluids (if used), and decontamination liquids, will be stored on site in appropriately labeled containers and disposed of properly. Disposable materials will be stored and disposed of separately. Containers must be labeled at the time of collection and will include date, location(s), site name, city, state, and description of matrix contained (e.g., soil, PPE). Waste will be managed in accordance with the *TGI – Investigation-Derived Waste Handling and Storage,* the procedures identified in the FIP/work plan or QAPP as well as

state-, federal- or client-specific requirements. Be certain that waste containers are properly labeled and documented in the field log.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Management of the original documents from the field will be completed in accordance with the site- specific QAPP.

In general, drilling activities will be documented on appropriate field/log forms as well as in a proper field notebook. All field data will be recorded digitally or with indelible ink. Field forms, logs/notes (including daily field and calibration logs), digital records, and chain-of-custody records will be maintained by the field team lead. Any deviations or omissions from this TGI should be documented.

Initial field logs and chain-of-custody records will be transmitted to the Arcadis CPM and Technical Lead at the end of each day unless otherwise directed by the CPM. The field teamleader retains copies of the field documentation.

Additionally, all documents (and photographs) will be scanned and electronically filed in the appropriate project directory for easy access. Pertinent information will include personnel present on site, times of arrival and departure, significant weather conditions, timing of drilling activities, soil descriptions, soil boring information, and quantities of materials used.

In addition, the locations of soil borings will be documented photographically and in a site sketch. If appropriate, a measuring wheel or engineer's tape will be used to determine approximate distances between important site features.

Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements.

11 Quality Assurance

Quality assurance procedures shall be conducted in accordance with the Arcadis Quality Management System or the site-specific QAPP.

All drilling equipment and associated tools (including augers, drill rods, sampling equipment, wrenches, and any other equipment or tools) that may have come in contact with soil will be cleaned in accordance with the procedures outlined in the appropriate TGI.

Field-derived quality assurance blanks will be collected as specified in the FIP/work plan and/or site- specific QAPP, depending on the project quality objectives. Typically, field rinse blanks (equipment blanks) will be collected when non-dedicated equipment (e.g., split-spoon sampler, stainless steel spoon) is used during soil sampling. Field rinse blanks will be used to confirm that decontamination procedures are sufficient and samples are representative of site conditions. Trip blanks for VOCs, which aid in the detection of contaminants from other media, sources, or the container itself, will be kept with the coolers and the sample containers throughout the sampling activities and during transport to the laboratory.

Operate all monitoring instrumentation in accordance with manufacturer's instructions and calibration procedures. Calibrate instruments at the beginning of each day and verify the calibration at the end of each day. Record all calibration activities in the field notebook.

12 References

ASTM D1586 - *Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils*. ASTM International. West Conshohocken, Pennsylvania.

13 Attachments

Attachment 1. Soil Boring Log Form

Attachment 1

Soil Boring Log Form

BORING LOG

ARCADIS

Boring ID:		**Project Name:**		**Page:**	
Permit ID:		**Date Started:**		**Ground Elevation:**	
Site Address:		**Date Completed:**		**Vertical Datum:**	
City, State:		**Total Depth:**		**Northing:**	
Drilling Co:		**Depth to Water:**		**Easting:**	
Driller:		**Hole Diameter:**		**Horizontal Datum:**	
Drilling Method:		**Core Device:**		**Prepared by:**	
Boring Status:		**Drilling Fluid:**		**Reviewed by:**	

Drilling Information				Primary Texture										Soil Description (Udden-Wentworth System)	Field Notes
				Fines		Sand					Gravel				
Drilling Depth (ft bgs)	Core Interval (ft)	Core Recovery (inches)	PID Reading (ppm)	clay	silt	very fine	fine	medium	coarse	very coarse	granule	pebble	cobble	boulder	Depth interval (ft), Moisture, PRIMARY TEXTURE, Modifier/Minor Texture, Sorting, Angularity, Consistency, Plasticity, Color - Only Record Sand Density with Standard Penetration Tests Minor Texture Modifiers: Trace (<10%), Little (10 to 20%), Some (21 to 35%), And (36 to 50%)	Driller's Observations, Particle Size Percentages, Geologic Formation, Field Screening Results, Sample Interval etc.

BORING LOG

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Boring ID: _____ **Project Name:** _____ **Page:** /

Drilling Information				Primary Texture											Soil Description (Udden-Wentworth System)	Field Notes
				Fines		Sand					Gravel					
Drilling Depth (ft bgs)	Core Interval (ft)	Core Recovery (inches)	PID Reading (ppm)	clay	silt	very fine	fine	medium	coarse	very coarse	granule	pebble	cobble	boulder	Depth interval (ft), Moisture, PRIMARY TEXTURE, Modifier/Minor Texture, Sorting, Angularity, Consistency, Plasticity, Color - Only Record Sand Density with Standard Penetration Tests Minor Texture Modifiers: Trace (<10%), Little (10 to 20%), Some (21 to 35%), And (36 to 50%)	Driller's Observations, Particle Size Percentages, Geologic Formation, Field Screening Results, Sample Interval etc.

BORING LOG

ARCADIS

Boring ID: _____ **Project Name:** _____ **Page:** /

Drilling Information				Primary Texture											Soil Description (Udden-Wentworth System)	Field Notes
				Fines		Sand					Gravel					
Drilling Depth (ft bgs)	Core Interval (ft)	Core Recovery (inches)	PID Reading (ppm)	clay	silt	very fine	fine	medium	coarse	very coarse	granule	pebble	cobble	boulder	Depth interval (ft), Moisture, PRIMARY TEXTURE, Modifier/Minor Texture, Sorting, Angularity, Consistency, Plasticity, Color - Only Record Sand Density with Standard Penetration Tests Minor Texture Modifiers: Trace (<10%), Little (10 to 20%), Some (21 to 35%), And (36 to 50%)	Driller's Observations, Particle Size Percentages, Geologic Formation, Field Screening Results, Sample Interval etc.

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TGI – Utility Locating Using Radio Frequency Methods

Rev: 4

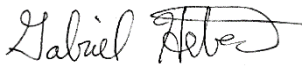
Rev Date: February 3, 2022

Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
Initial Draft	001	March 2017	N/A	N/A	Grant Sprick
Review	002	March 22, 2017	N/A	See tracked changes	Brent Barker
Final Draft/Signatures	003	October 12, 2018	All	Modification to TGI format	Gregory Byer Brent Barker
Revision/ Signatures	004	February 3, 2022	All	Update of Technical Guidance	Gabriel Hebert Gregory Byer

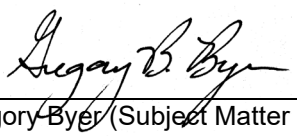
Approval Signatures

Prepared by: 2/3/2022



Gabriel Hebert (Preparer) Date

Reviewed by: 2/3/2022



Gregory Byer (Subject Matter Expert) Date

1 Introduction

This Technical Guidance Instruction (TGI) is intended to provide general guidance on the use of Radio Frequency Locators (RFLoc) for the location and tracing of buried utility lines. Radiodetection (2007, 2017) and Schonsteadt (2002) provides technical background regarding the applicability of RFLoc. This guidance is intended to be used to provide Arcadis CPMs and technical staff with a uniform set of procedures either in cases where Arcadis is subcontracting the data collection and processing or in circumstances where the CPM and affiliated technical staff are participating in a RFLoc survey led by designated Arcadis geophysical specialists. Whether the data collection and processing are being subcontracted or performed in-house, directed by geophysical specialists, in all cases the non-geophysical CPM and/or technical staff should consult with Arcadis geophysical specialists for survey design and scoping, data management and processing, and proper data interpretation procedures. It should be noted that it is commonplace to perform RFLoc surveys as a part of a larger scope of work that may include other geophysical techniques such as seismic, electrical resistivity, magnetics, electromagnetics as well as direct observation (ground truthing) with drilling and borehole geophysics.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

This reference document outlines the applications, limitations, and methodology associated with the use of Radio Frequency Locators (RFLoc). RFLoc methods can be active, utilizing a radio frequency (RF) transmitter that applies a known frequency to an underground utility and a receiver that is able to detect this frequency along the length of the structure, or passive, utilizing just the receiver to trace pre-existing or ambient RF signals being transmitted along the utility.

RFLoc methods can be useful in tracing buried metallic utility lines, or non-metallic utility lines that were installed in conjunction with a metallic tracer wire. In passive tracing, direct contact with the utility in question is not required and a transmitter is not necessary, as the **receiver** takes advantage of signals that are already present on buried metallic conductors – such as active power, radio signals from ambient radio towers, and cathodic protection systems (CPS). In active tracing, the RF signal transmission is applied to the utility line either by direct connection or through signal induction. In the Direct Connection Method, the signal is applied to an exposed segment of the utility of concern (or associated tracer wire), such as an exposed conduit or segment of pipe, using the provided alligator clip connectors or a coupling ring. In the Signal Induction Method, the transmitter is placed in the vicinity of and in-line with the anticipated direction of the utility in questions, and “induces” a RF signal in the buried line. Electrically conductive lines will “carry” or transmit the RF signal, which can then be traced from the ground surface using the receiving unit.

Examples of conductive lines commonly traced include telephone, electric, and water main utility lines. RFLoc is typically not effective in tracing non-conductive lines, including materials composed of PVC, fiberglass, concrete, or clay, unless a tracer-wire to which an RF signal can be applied was installed with the non-conductive line. Additionally, in certain cases, a sonde transmitter can be advanced and traced through non-conductive lines as well.

RFLoc methods are non-invasive and non-destructive. These tools transmit and receive radio frequency electromagnetic signals. Subsurface objects or material contacts can be located based on differences in their dielectric potential and/or electrical resistivity. Important constraints to consider are depth of signal penetration, resolution, and signal transmission. Higher frequency signals will yield better resolution but offer less depth of penetration and range of transmission as compared to a lower frequency signals. Additionally, signal transmission can be dampened out or limited by carry-over onto other utilities or lack of a good “connection” due to physical constraints.

4 Personnel Qualifications

Utility locating/tracing investigations should be conducted by qualified and experienced operators, such as an experienced field technician and/or a geophysicist. The RFLoc operator should be experienced in the use of the instrument and its limitations. The operator should be able to adjust data acquisition procedures in response to variable site conditions to identify anomalies and resolve target features. Inexperienced personnel directing or supervising RFLoc investigations have the responsibility to seek appropriate guidance, training, and technical peer review from qualified and experienced personnel prior to use.

In accordance with the Utility Location SOP, field personnel conducting subsurface work and/or investigation (SWI) activities where above ground or underground utilities are in the vicinity of the work have the responsibility to read, understand, and follow the ***Utility Location Policy and Procedure***. This procedure requires that any personnel assisting in the identification of underground utilities need to have previous related experience of a minimum of 1 year. Those implementing remote sensing technologies must complete training in those techniques and have 6 months experience operating and interpreting results. If utilities cannot be located to eliminate any reasonable concern, field personnel can use their Stop Work authority until utility locations can be properly identified. Field personnel must review this procedure onsite with subcontractors, and ensure they follow the

procedures detailed in this document. Any Arcadis subcontractor not following the Utility Location Policy and Procedures will be asked to stop work, and the project manager will be contacted.

5 Equipment List

The following equipment will be available, as required, during RFLoc utility locating surveys.

- Personal protective equipment (PPE), as required in the site Health and Safety Plan (HASP);
- Appropriate forms, Site plans/maps, field notebook, spray paint and camera;
- RF Cable, Pipe, Utility Locator transmitter and receiver;
- Non-conductive measuring tape; and
- Spare batteries (typically 6 to 8 x C or D-sized alkaline).

6 Cautions

The effectiveness of locating buried utilities is site-specific and subject to the skill level of the operator. Reliability and efficiency are enhanced when used in conjunction with other lines of evidence, including other geophysical methods, detailed reconnaissance of the area, review of available site plans, and overall understanding of the Site area and types of buried utilities that may be expected at the Site.

RF signal interferences can also occur. Utilities that are located in proximity to another utility line, overlapping, or in layers may appear as one unit. In some cases, segments of lines being traced may have been previously damaged/severed or partially removed and capped, which may cause an abrupt loss of signal transmission. RF transmission signals can also “jump” from one utility line to another, particularly when higher frequencies are used. Signals will generally follow the path of least resistance, and in some instances, will deviate from the line of interest or cause a shadow zone where one or more utilities are located in close proximity.

Also, as noted above, non-metallic and small diameter utilities are not conducive to locating using RFLoc methods. However, in these cases, alternative methods can be considered, such as tracing a transmitting lead (also known as a sonde or “mouse”), which can in some cases be snaked into a non-conductive pipe and traced at the surface using the receiving unit.

7 Health and Safety Considerations

Minimize physical hazard exposure through use of proper PPE as prescribed in the HASP. Maintain awareness of other potential hazards associated with the physical location where the RFLoc investigation is being conducted and any ingress or egress conditions. Special care should always be used whenever a direct connection is being made to a buried cable, or when applying an induction loop to an exposed wire or cable. Never physically touch or connect to the equipment to exposed wires or cables that may carry electricity. Never connect directly to live or energized power cables.

8 Procedure

1. Become familiar with the details of the applicability and limitations of the RF line locating equipment that has been selected for the project. Most commercially available RFLoc equipment have similar operating functions, although operating menus and selections will vary from one model to another. Users must read all operating instructions and become familiar with any instrument prior to using the instrument to locate utilities.

2. Evaluate site-specific utility information to determine suitability of the equipment (e.g., depth, diameter, age, and conductivity of the utilities).

3. Evaluate the Site conditions and select one or more of the potential signal transmission methods for optimal use, including passive, direct connections, transmitter clamp, and/or inductive transmission.

4. **Passive Method:** This is the easiest method of RF line locating, as direct contact with the utility in question is not required and a transmitter is not necessary. The operator simply uses the **receiver** to trace signals that are already present on buried metallic conductors – such as active power, radio signals from ambient radio towers, and cathodic protection systems (CPS). Active AC power lines transmitting at either 50 or 60 Hz will be detected, as will CPS lines operating at 100 or 120 Hz. Additionally, metallic utility lines in relative proximity to radio towers will have a small current induced in them, which can be traced in passive mode. To trace in passive mode, either select RADIO mode to search for induced signals form radio towers, or POWER mode to search for power or CPS lines.

5. **Direct Connection Method:** This is the most reliable method of RF signal application, as it is relatively free of interference. Additionally, the greatest amount of signal strength can be achieved by this method. Low, mid, and high frequency signals may be used. To trace utility lines with this method, connect the RED test cord to an existing exposed metallic section of the utility (or tracer wire) via the alligator clip. If the RED test cord will not fit on the desired utility, a metal clamp can be attached to the utility, and the RED test cord may be attached to the metal clamp. Next, place the metal GROUNDING ROD approximately 10 feet (or as far as possible) from this point, at an angle of 90º to the anticipated direction of the buried cable or pipe. Push the GROUNDING ROD into the ground 8 to 10 inches (or as far as possible). Connect the BLACK test cord to the GROUNDING ROD via the alligator clip. Plug the combined RED/BLACK test cords into the transmitter and turn it on.

An alternative to using the RED/BLACK test cords is to the BLACK TRANSMITTER CLAMP, especially for utility lines that are too large and/or difficult to affix the alligator clips to. It simply fits around a pipe or cable and safely applies an active signal to a utility without interrupting or disconnecting the supply. However, successful coupler operation requires an insulated conductor that is grounded on both near and far ends. To use, put the transmitter clamp around the cable and ensure the clamp jaws are closed. For best results, try to connect the clamp around utilities furthest away from the system ground. The result will be a stronger signal. By connecting near the grounding, the range will also be shorter, and difficulty may arise when trying to determine one cable from another. Then, plug the transmitter clamp cord into the transmitter.

Next, press the FREQUENCY button to select the desired frequency (e.g., 815 Hz up to 200 kHz). The Power Output Indicator and the Frequency light of the chosen frequency will light up. Select strength of the signal to be applied to the utility line. Stronger signals travel further but can jump to adjacent conductors. Weaker signals do not travel as far but are less likely to transfer to other conductors. The utility can now be traced.

6. **Signal Induction Method:** This method is convenient to use, as direct contact is not needed with the utility in question, and services are not interrupted. However, to work properly, the utility in question must have good insulation or a non-conductive coating, or the operating range will be short. The use of higher frequencies is recommended in induction mode, as they are induced easier onto nearby utilities. To use the signal induction method, turn the transmitter on, and select a higher frequency. Place the transmitter as close as possible to the anticipated location of the cable or pipe and in line with the anticipated alignment of the cable or pipe. The utility can now be traced.

7. **Sonde Transmitter Methods:** A sonde transmitter is a small battery powered signal transmitter that is typically attached to semi-rigid tooling (such as a sewer “snake”) and inserted into non-metallic ducts, drains or sewers so they can be located and traced with a receiver at the surface. A wide range of transmitting sondes are available to suit different applications. Sondes can also be used to pinpoint joints in iron gas pipes, locate blockages in plastic water pipes, and monitor the progress of horizontal boring tools. Sondes may also be floated along drains at the end of a tether, and floats are available for fitting to the sewer sonde. Sondes can be strapped to high-pressure water jets or similar devices used for cleaning,

maintaining, and inspecting drains. Tracing the sonde along the pipe can then be accomplished by activating the transmitter and following the sonde with the receiver from the surface. Care must be used to ensure the sonde does not become snagged, which may prevent its recovery from the pipe.

8. **Selecting the Tracing Signal Frequency:** The choice of signal frequency (typically 815 Hz through 200 kHz) is dependent on the Site conditions, with each having their advantages. It is recommended to begin by using the lowest available instrument signal frequency (e.g., 815 Hz) and continue as long as you are confident in the results. If the signal is very weak, try to adjust the connection or grounding. If there is no improvement in signal, then try increasing the frequency. Repeat adjustments of ground and connection point again until switching to the highest frequency. A lower frequency signal is usually preferred to the mid-range and high frequency signal, because it is much less susceptible to locating errors caused by nearby cables or pipes. Higher frequencies work better in the signal induction method.

The lower frequency locating range will also allow for longer line traces than the higher frequency signal and will not travel well through disconnected shield bonds or insulated pipe bushings. The mid-range frequency averages the best of both high and low frequency and is not as susceptible to bleed off or coupling, but it can “jump” from one utility to another. As such, it is still best to use the lower frequency, when possible, but the mid-range is commonly used to locate coaxial cable and telecom pairs. The higher frequency is sometimes better for locating sharp corners in cables or pipes, where higher resolution location is necessary, although the locating range is quite short for the high frequency signal, so the transmitter must be repositioned more often during the tracing operation.

9. **Null and Peak Modes:** Most RFLoc instruments allow the Operator to select between NULL and PEAK modes. PEAK mode provides the most sensitive and accurate mode for location and depth measurement, and as such, it should be the mode selected in most circumstances. It provides a sharp peak response over the center of the utility, with a corresponding small decrease in sensitivity as the receiver is moved to either side. NULL mode gives a null response when it is directly over the line. The sharp, null response is easier to use than the peak response but is vulnerable to interference and should not be purely used for locating (more for confirmation of the Peak mode), except in areas where there is no interference present.

10. **Tracing Utility Lines:** Once a tracing method (active or passive), signal transmission method (Direct, Induction, or Sonde), mode (PEAK or NULL), and desired frequency has been selected, the utility in question can now traced. To trace a utility, start at the location of your transmitter and make a full circle around your transmitter, noting where the receiver response (either PEAK or NULL) indicates the presence of a utility line. Once the general orientation of the utility line has been noted and marked, step out gradually in that direction, sweeping the receiver in 180-degree arcs, marking the location and depth of the utility as you go, being aware of potential bends in the line. For both Direct and Induction transmission methods, the transmission signal will gradually attenuate the further from the transmitter the receiver is moved, or if the signal is transmitting through numerous utility intersections or jumping to numerous adjacent utilities. For long utility alignments, utilizing a higher transmission power output or lower frequencies can extend the range of the transmitted signal. If the transmission signal is still unable to be detected at longer distances, a secondary location for the transmitter can be selected. For the Induction method, this simply involves moving the transmitter to a point 25 feet behind the last strong signal and continue searching. For the Direct method, this involves attempting to find another location further down the utility alignment where the utility is accessible from the ground surface.

11. Consider complimentary technologies to supplement RFLoc methods and provide multiple lines of evidence. Such technologies may include Ground Penetrating Radar (GPR), magnetic and/or electromagnetic surveys, and air-knifing or vacuum explorations.

12. Only employ qualified and experienced RFLoc operators. For utility locating and mapping applications, the RFLoc operator should be specifically experienced in evaluating data quality, as well as identifying anomalies in the field requiring variations in data acquisition procedures in order to positively interpret and locate targets of concern.

13. Complete appropriate mark-outs at the Site, such as spray painting and/or flagging traced lines. Consideration in the usage of paint colors should also be given. Standard colors for utility mark outs include: **RED-Electric: YELLOW-Gas; ORANGE-Communication: BLUE-Water; GREEN-Sewer and Drain; PURPLE-Reclaimed Water; PINK-Temporary Survey Marking; WHITE- Proposed Excavation.**

9 Waste Management

RFLoc is a non-invasive procedure and should not result in the generation of derived wastes. Any trash or rubbish generated during the course of field activities should be disposed of in a proper trash receptacle.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Should FieldNow® applications not have the capabilities to fully digitally capture the post survey site conditions, then field notes and site photos should be taken to include a description of areas surveyed, areas that were not surveyed, interferences, equipment and methods used, location and type of utilities located (if known), and a final site sketch should be completed if possible. Taped distance measurements should be taken to marked lines relative to existing/permanent site features for future reference. Project team personnel shall be responsible for maintaining all mark outs for the duration of the project, or have the lines remarked if lost due to weather and/or site activities.

11 Quality Assurance

The following quality control procedures should be observed:

- Seek appropriate input prior to conducting an RFLoc survey to identify site-specific features that may impact data acquisition.
- Operate all equipment in accordance with manufacturer's instructions and recommended procedures. Record all system component information in the field book.
- Data quality should be checked in the field to identify anomalies that may require adjustment to the data acquisition procedures. Make appropriate adjustments to data acquisition methods to achieve survey objectives, as feasible.
- If possible, it is recommended that the operation and performance of the RFLoc equipment be field checked by locating existing underground utilities or structures of known depth, size, and construction. These characteristics should be similar to that of unidentified target objects.
- Data interpretation should undergo peer review by appropriate qualified and experienced personnel.

12 References

Radiodetection.com (2007). RD7100 Cable and Pipe and Pipe Locator User Guide.PDF

Radiodetection.com (2017). The Theory of Buried Cable and Pipe Location.PDF

Schonstedt.com (2002). U-Locate Pipe and Cable Locator User Guide.PDF

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ARCADIS INSTRUMENT CALIBRATION LOG

Project: Fort Leavenworth PA/SI

Project Number: 02118216.1000.8AA00

Personnel:
Kenneth (Wes) Kuhns, Jacob Creighton

Water Quality Instrument: YSI 600 MPS — **Serial Number:**

Parameter	Date		Date		Date		Date		Date		Date		Date	
	Initial Read	Calibrated to	Initial Read	Calibrated to	Initial Read	Calibrated to	Initial Read	Calibrated to	Initial Read	Calibrated to	Initial Read	Calibrated to	Initial Read	Calibrated to
ph 4														
ph 7														
ph 10														
Conductivity 1.413 mS/cm														
ORP 240 mV														
Temperature	-	-	-	-	-	-	-	-	-	-	-	-	-	-
DO	-	-	-	-	-	-	-	-	-	-	-	-	-	-

Turbidity Meter Instrument: Hach 2100Q — **Serial Number:**

Parameter	Date		Date		Date		Date		Date		Date		Date	
	Initial Read	Calibrated to	Initial Read	Calibrated to	Initial Read	Calibrated to	Initial Read	Calibrated to	Initial Read	Calibrated to	Initial Read	Calibrated to	Initial Read	Calibrated to
Turbidity 10 NTU	-	(verification)	-	(verification)	-	(verification)	-	(verification)	-	(verification)	-	(verification)	-	(verification)
Turbidity 20 NTU		20		20		20		20		20		20		20
Turbidity 100 NTU		100		100		100		100		100		100		100
Turbidity 800 NTU		800		800		800		800		800		800		800

Photoionization Detector Instrument: qRae 4-gas Meter — **Serial Number:**

Standard	Date		Date		Date		Date		Date		Date		Date	
	Standard Concentration	Meter Reading	Standard Concentration	Meter Reading	Standard Concentration	Meter Reading	Standard Concentration	Meter Reading	Standard Concentration	Meter Reading	Standard Concentration	Meter Reading	Standard Concentration	Meter Reading

Document Control Number:TGM - _____
TGM + project number plus date as follows: xxxxxxxx.xxxx.xxxxx - dd/mm/year

TAILGATE HEALTH & SAFETY MEETING FORM

This form documents the tailgate meeting conducted in accordance with the Project HASP. Personnel who perform work operations on-site during the day are required to attend this meeting and to acknowledge their attendance, at least daily.

Project Name:			**Project Location:**
Date:	**Time:**	**Conducted by:**	**Signature/Title:**
Client:		**Client Contact:**	**Subcontractor companies:**

TRACKing the Tailgate Meeting

Think through the Tasks (list the tasks for the day):

1 _____ 3 _____ 5 _____

2 _____ 4 _____ 6 _____

Other Hazardous Activities - Check the box if there are any other Arcadis, Client or other party activities that may pose hazards to Arcadis operations ☐ If there are none, write "None" here: _____

If yes, describe them here: _____

How will they be controlled? _____

Prework Authorization - check activities to be conducted that require permit issuance or completion of a checklist or similar before work begins:

	Doc #		Doc #		Doc #
☐ Not applicable		☐ Working at Height		☐ Confined Space	
☐ Energy Isolation (LOTO)		☐ Excavation/Trenching		☐ Hot Work	
☐ Mechanical Lifting Ops		☐ Overhead & Buried Utilities		☐ Other permit	

Discuss following questions (for some review previous day's post activities). **Check if yes :**

☐ Incidents from day before to review?	☐ Lessons learned from the day before?	☐ Any Stop Work Interventions yesterday?
☐ Any corrective actions from yesterday?	☐ Will any work deviate from plan?	☐ If deviations, notify PM & client
☐ JSAs or procedures are available?	☐ Field teams to "dirty" JSAs, as needed?	☐ All equipment checked & OK?
☐ Staff has appropriate PPE?	☐ Staff knows Emergency Plan (EAP)?	☐ Staff knows gathering points?

☐ Topics from Corp H&S to cover?

Comments: _____

Recognize the hazards (check all those that are discussed) (Examples are provided) and **Assess** the Risks (Low, Medium, High - circle risk level) - Provide an overall assessment of hazards to be encountered today and briefly list them under the hazard category.

☐ Gravity (i.e., ladder, scaffold, trips) (L M H)	☐ Motion (i.e., traffic, moving water) (L M H)	☐ Mechanical (i.e., augers, motors) (L M H)
☐ Electrical (i.e., utilities, lightning) (L M H)	☐ Pressure (i.e., gas cylinders, wells) (L M H)	☐ Environment (i.e., heat, cold, ice) (L M H)
☐ Chemical (i.e., fuel, acid, paint) (L M H)	☐ Biological (i.e., ticks, poison ivy) (L M H)	☐ Radiation (i.e., alpha, sun, laser) (L M H)
☐ Sound (i.e., machinery, generators) (L M H)	☐ Personal (i.e. alone, night, not fit) (L M H)	☐ Driving (i.e. car, ATV, boat, dozer) (L M H)

Continue TRACK Process on Page 2

TAILGATE HEALTH & SAFETY MEETING FORM - Pg. 2

Control the hazards (Check all and discuss those methods to control the hazards that will be implemented for the day): Review the HASP, applicable JSAs, and other control processes. Discuss and document any additional control processes.

- [x] **STOP WORK AUTHORITY** (Must be addressed in every Tailgate meeting - (*See statements below*)

☐ Elimination	☐ Substitution	☐ Isolation
☐ Engineering controls	☐ Administrative controls	☐ Monitoring
☐ General PPE Usage	☐ Hearing Conservation	☐ Respiratory Protection
☐ Personal Hygiene	☐ Exposure Guidelines	☐ Decon Procedures
☐ Emergency Action Plan (EAP)	☐ Fall Protection	☐ Work Zones/Site Control
☐ JSA to be developed/used ***(specify)***	☐ TIP conducted ***(specify job/JSA)***	☐ Traffic Control
☐	☐	☐ Other ***(specify)***
☐	☐	☐

Signature and Certification Section - Site Staff and Visitors

Name/Company/Signature	Initial & Sign in Time	Initial & Sign out Time	I have read and understand the HASP

Important Information and Numbers

All site staff should arrive fit for work. If not, they should report to the supervisor any restrictions or concerns.

In the event of an injury, employees will call WorkCare at 1.800.455.6155 and then notify the field supervisor who will then notify the Project or Task Manager.

In the event of a motor vehicle accident, employees will notify the field supervisor who will then notify the Project or Task Manager.

In the event of a utility strike or other damage to property of a client or 3rd party, employees will immediately notify the field supervisor, who will then immediately notify the Project or Task Manager.

Visitor Name/Co - not involved in work

	In	Out
	In	Out
	In	Out
	In	Out

I will STOP the job any time anyone is concerned or uncertain about health & safety or if anyone identifies a hazard or additional mitigation not recorded in the site, project, job or task hazard assessment.

I will be alert to any changes in personnel, conditions at the work site or hazards not covered by the original hazard assessments.

If it is necessary to **STOP THE JOB,** I will perform **TRACK**; and then amend the hazard assessments or the HASP as needed.

I will not assist a subcontractor or other party with their work unless it is absolutely necessary and then only after I have done TRACK and I have thoroughly controlled the hazard.

Post Daily Activities Review - Review at end of day or before next day's work (Check those applicable and explain:)

- ☐ Lessons learned and best practices learned today: _____
- ☐ Incidents that occurred today: _____
- ☐ Any Stop Work interventions today? _____
- ☐ Corrective/Preventive Actions needed for future work: _____
- ☐ Any other H&S issues: _____

Keep H&S 1st in all things

WorkCare - 1.800.455.6155

ARCADIS

Low-flow Groundwater Purging and Sampling Form

Project No. _____ Well ID: _____ Date: _____

Project Name/Location: _____ Personnel: _____ Weather: _____

Measuring Pt. Description: _____ Screen Setting (ft-btoc): _____ Casing Diameter (in.): _____ Well Material: _____ PVC _____ SS

TOC Elevation: _____ Total Depth As Built (ft bgs): _____ Sample Method: _____

Static Water Level (ft-btoc): _____ Measured (ft btoc): _____ Pump Intake (ft-btoc): _____ Purge Method: _____

Pump On: _____ Volume Purged (L): _____

Water Quality Meter Make/ Model: _____

Stabilization parameters 3 readings:

Time (approx. 3-5 minute interval)	Minutes Elapsed	Rate <250 (mL/min)	Depth to Water (Δ not to exceed 0.3 ft or <10% of sat. screen) (ft btoc)	Liters Purged	3% Temp. (ºC)	3% Conductivity (mS/cm)	10% Dissolved Oxygen (mg/L)	0.1 pH (S.U.)	10 mV ORP (mV)	10%, w'in 1 NTU if <10 Turbidity (NTU)	Appearance	
											Color	Odor

Sample ID: _____

	Y	N
MS/MSD:	☐	☐
Duplicate:	☐	☐
Equipment Blank:	☐	☐

Duplicate ID: _____ Time: _____

Equipment Blank ID: _____ Time: _____

Sample Time: Label _____ Start _____ End/Pump Off _____

Pump Settings: _____

Notes: _____

Constituents sampled	**Container:**	**Number:**	**Preservative^**	**Collected?**
PFAS Group*	500 mL HDPE w/ HDPE screw cap		--	☐
				☐
				☐
				☐
				☐
				☐
				☐
				☐
				☐
#REF!	#REF!			Total:

Gallons/Foot: 1" = 0.04; 1.25" = 0.06; 1.5" = 0.09; 2" = 0.16; 2.5" = 0.26; 3" = 0.37; 3.5" = 0.50; 4" = 0.65; 6" = 1.47

Well and Location Information

Condition of Surrounding Area: _____ Well Lock Condition: _____

Condition of Well: _____ Well Locked at Departure: Yes / No

Well Completion: _____

Completed By: _____ Date: _____ Reviewed By: _____ Date: _____

ON THE ORIGINAL DRAWING.
USE TO VERIFY FIGURE
REPRODUCTION SCALE

SOIL DESCRIPTION FIELD GUIDE (JUNE 30, 2017; REV. 2.0)

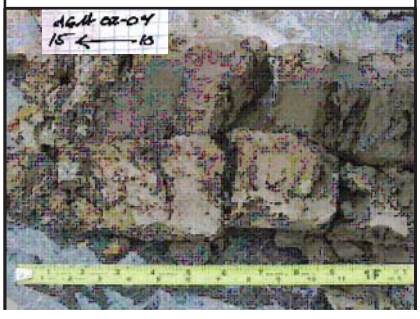
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FINE-GRAINED SOILS

Description	Criteria
	Descriptor - Plasticity
Nonplastic	A 1/8-inch (3mm) thread cannot be rolled at any moisture content.
Low	Thread can barely be rolled, and lump cannot be formed when drier than plastic limit.
Medium	Takes considerable time and rolling to reach plastic limit. Thread cannot be rolled after reaching plastic limit. Lump crumbles when drier than plastic limit.
High	Thread is easy to roll and quickly reaches plastic limit. Thread can be rerolled several times after reaching plastic limit. Lump can be formed without crumbling when drier than plastic limit.
	Descriptor - Dilatancy
No Dilatancy	No visible change when shaken or squeezed.
Slow	Water appears slowly on the surface of soil during shaking and does not disappear or disappears slowly when squeezed.
Rapid	Water appears quickly on surface of soil during shaking and disappears quickly when squeezed.
Minor Components with Descriptors	
	Moisture
Dry	Absence of moisture, dry to touch, dusty.
Moist	Damp but no visible water.
Wet	Visible free water; soil is usually below the water table. (Saturated)
	Consistency
Very soft	N-value < 2 or easily penetrated several inches by thumb.
Soft	N-value 2-4 or easily penetrated 1 inch by thumb.
Medium stiff	N-value 5-8 or indented about 1/2 inch by thumb with great effort.
Stiff	N-value 9-15 or indented about 1/4 inch by thumb with great effort.
Very stiff	N-value 16-30 or readily indented by thumb nail.
Hard	N-value > than 30 or indented by thumbnail with difficulty.
Color using Munsell	
Geologic Origin (if known)	
Other	

EXAMPLE OF SOIL DESCRIPTION AND PHOTO

10-15 feet CLAY, medium to high plasticity; trace silt; trace small to very large pebbles, subround to subangular up to 2" diameter; moist, stiff, dark grayish brown (10YR 4/2). NOTE: Lacustrine; laminated 0.1 to 0.2" thick, laminations brownish yellow (10YR 4/3).



DESCRIPTION ORDER

Depth Interval
Principal Components with Descriptors
Minor Components with Descriptors
Sorting
Field Moisture Condition
Density/Consistency
Color using Munsell
Geologic Origin (if known)
Other descriptions as NOTES:
- Odor
- Stratigraphy
- Structure
- Sphericity
- Cementation
- Reaction to acid

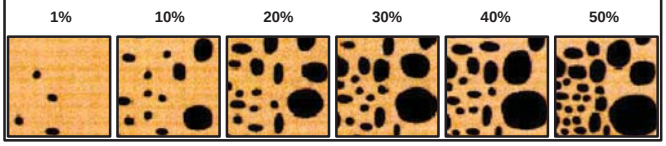
MINOR COMPONENTS % MODIFIERS

Modifier	Percent of Total Sample (by volume)
and	36 - 50
some	21 - 35
little	10 - 20
trace	<10

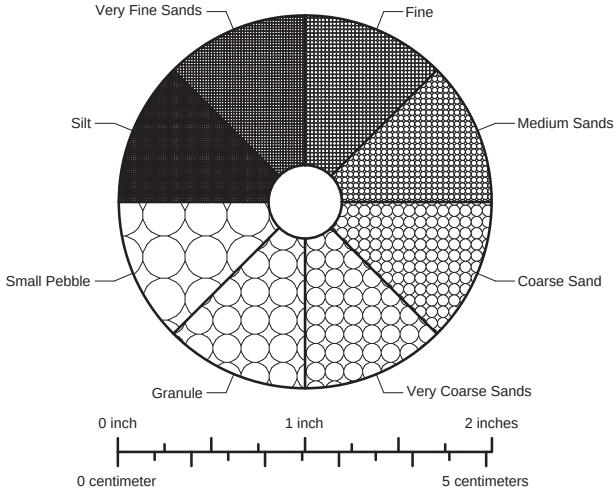
UDDEN-WENTWORTH SCALE

Fraction	Sieve Size	Grain Size	Approximate Scale
Boulder		256 - 4096 mm	Larger than volleyball
Large Cobble		128 - 256 mm	Softball to volleyball
Small Cobble		64 - 128 mm	Pool ball to softball
Very Large Pebble		32 - 64 mm	Pinball to pool ball
Large Pebble		16 - 32 mm	Dime size to pinball
Medium Pebble		8 - 16 mm	Pencil eraser to dime size
Small Pebble	No. 5+	4 - 8 mm	Pea size to pencil eraser
Granule	No. 10 - 5	2 - 4 mm	Rock salt to pea size
Very Coarse Sand	No. 18 - 10	1 - 2 mm	See field gauge card
Coarse Sand	No. 35 -18	0.5 - 1 mm	See field gauge card
Medium Sand	No. 60 - 35	0.25 - 0.5 mm	See field gauge card
Fine Sand	No. 120 - 60	0.125 - 0.25 mm	See field gauge card
Very Fine Sand	No. 230 - 120	0.0625 - 0.125 mm	See field gauge card
Silt and Clay. See SOP for description of fines	Not Applicable	<0.0625 mm	Analyze by pipette or hydrometer

PARTICLE PERCENT COMPOSITION ESTIMATION



GRAPH FOR DETERMINING SIZE OF PARTICLES



FOR COARSE-GRAINED SOILS

Description	Criteria
	Descriptor - Angularity
Angular	Particles have sharp edges and relatively planar sides withunpolished surfaces.
Subangular	Particles are similar to angular but have rounded edges.
Subround	Particles have nearly planar sides but have well-roundedcorners and edges.
Round	Particles have smoothly curved sides and no edges.
Minor Components with Descriptors	
	Sorting Cu= d60/d10
Well Sorted	Near uniform grain-size distribution Cu= 1 to 3.
Poorly Sorted	Wide range of grain size Cu= 4 to 6.
	Moisture
Dry	Absence of moisture, dry to touch, dusty.
Moist	Damp but no visible water.
Wet	Visible free water; soil is usually below the water table. (Saturated)
	Density
Very loose	N-value 1 - 4
Loose	N-value 5 - 10
Medium Dense	N-value 11 - 30
Dense	N-value 31 - 50
Very dense	N-value >50
Color using Munsell	
Geologic Origin (if known)	
Other	
	Cementation
Weak Cementation	Crumbles or breaks with handling or little finger pressure.
Moderate Cementation	Crumbles or breaks with considerable finger pressure.
Strong Cementation	Will not crumble with finger pressure.
	Reaction with Dilute HCl Solution (10%)
No Reaction	No visible reaction.
Weak Reaction	Some reaction, with bubbles forming slowly.
Strong Reaction	Violent reaction, with bubbles forming immediately.

EXAMPLE OF SOIL DESCRIPTION AND PHOTO

10 -15 feet SAND, medium to very coarse; little granules to medium pebbles, subround to subangular; trace silt; poorly sorted, wet, grayish brown (10YR5/2).



ON THE ORIGINAL DRAWING.
USE TO VERIFY FIGURE
REPRODUCTION SCALE

SOIL DESCRIPTION FIELD GUIDE (JUNE 30, 2017; REV. 2.0)

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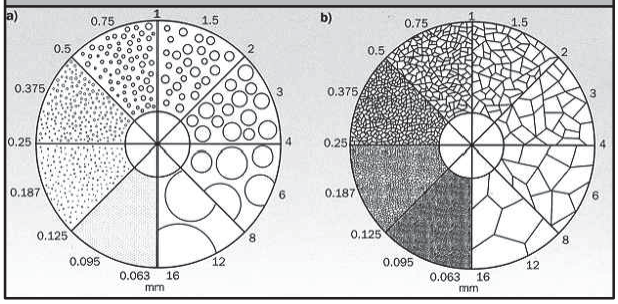
VARIATIONS IN SOIL STRATIGRAPHY

Term	Thickness of Configuration
Parting	0 - to 1/16-inch thickness.
Seam	1/16 - to 1/2-inch thickness.
Layer	1/2 - to 12-inch thickness.
Stratum	> 12-inch thickness.
Pocket	Small erratic deposit, usually less than 1 foot in size.
Varved Clay	Alternating seams or layers of sand, silt, and clay (laminated).
Occasional	≤ 1 foot thick.
Frequent	> 1 foot thick.

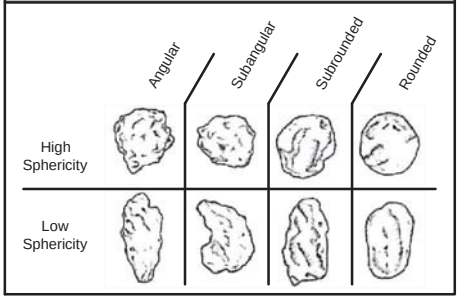
SOIL STRUCTURE DESCRIPTIONS

Term	Description
Homogeneous	Same color and appearance throughout.
Laminated	Alternating layers < 1/4 inch thick.
Stratified	Alternating layers ≥ 1/4 inch thick.
Lensed	Inclusions of small pockets of different materials, such as lenses of sand scattered through a mass of clay; note thickness.
Blocky	Cohesive soil can be broken down into small angular lumps, which resist further breakdown.
Fissured	Breaks along definite planes of fracture with little resistance to fracturing.
Slickensided	Fracture planes appear to be polished or glossy, sometimes striated.

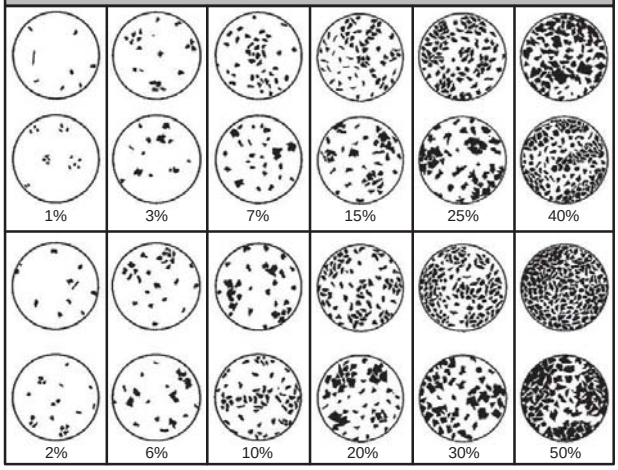
GRAPH FOR DETERMINING SIZE OF PARTICLES



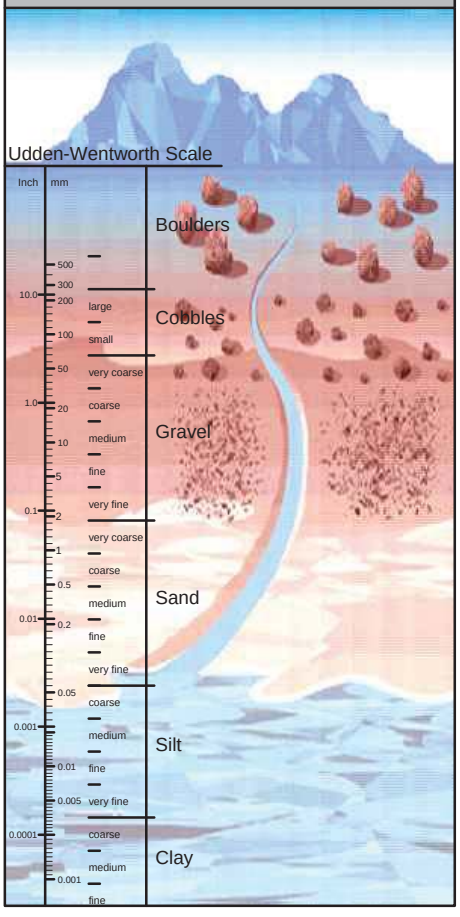
ANGULARITY CHART



PARTICLE PERCENT COMPOSITION ESTIMATION



SORTING



SETTLING TABLE (SILT/CLAY)

Diameter of Particle (mm)	<0.625	<0.031	<0.016	<0.008	<0.004	<0.002	<0.0005
Depth of Withdrawal (cm)	10	10	10	10	5	5	3
Time of Withdrawal	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec	hr:min:sec
Temperature (Celsius)							
20	00:00:29	00:01:55	00:07:40	00:30:40	00:61:19	04:05:00	37:21:00
21	00:00:28	00:01:52	00:07:29	00:29:58	00:59:50	04:00:00	
22	00:00:27	00:01:50	00:07:18	00:29:13	00:58:22	03:54:00	
23	00:00:27	00:01:47	00:07:08	00:28:34	00:57:05	03:48:00	
24	00:00:26	00:01:45	00:06:58	00:27:52	00:55:41	03:43:00	33:56:00
25	00:00:25	00:01:42	00:06:48	00:27:14	00:54:25	03:38:00	
26	00:00:25	00:01:40	00:06:39	00:26:38	00:53:12	03:33:00	
27	00:00:24	00:01:38	00:06:31	00:26:02	00:52:02	03:28:00	
28	00:00:24	00:01:35	00:06:22	00:25:28	00:50:52	03:24:00	31:00:00
29	00:00:23	00:01:33	00:06:13	00:24:53	00:49:42	03:10:00	
30	00:00:23	00:01:31	00:06:06	00:24:22	00:48:42	03:05:00	

10 inches
9 inches
8 inches
7 inches
6 inches
5 inches
4 inches
3 inches
2 inches
1 inch
1/10in

0 mm
10 mm
20 mm
30 mm
40 mm
50 mm
60 mm
70 mm
80 mm
90 mm
100 mm
110 mm
120 mm
130 mm
140 mm
150 mm
160 mm
170 mm
180 mm
190 mm
200 mm
210 mm
220 mm
230 mm
240 mm
250 mm

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Boring ID: _____

Field Boring Log

Client:	Field Location:	**Boring ID:**	**Date:**	
Project No.:		Latitude:	Longitude:	Ground Surface Elevation:
Site:		Drilling Method:	Drill Rig Model:	
Logged by:		Soil Sampling Method:	Groundwater Sampling Method:	
Drilling Co.:		Hole diameter:	Total depth of boring:	Reference Datum:
Driller:		Depth to first encountered water:		

Time	Drill Notes	Depth (ft bgs)	Soil/Rock Description (principal components and angularity; minor components and angularity; sorting; moisture content; consistency/density; color; additional comments)	Soil Sample	Groundwater Sample	Sample IDs and Notes

Appendix B

Laboratory Certification and Standard Operating Procedures

Quality Assurance Manual Cover Page

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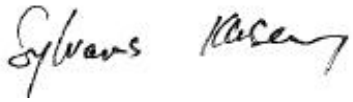
Quality Assurance Manual
Approval Signatures



Laboratory Director/Business Unit Manager-Mark Acierno — 09/13/2022 Date



Quality Assurance Manager - Carl Armbruster — 09/13/2022 Date



Technical Manager, Organics – Sylvanus Klusey — 09/13/2022 Date



Technical Manager, Inorganics – Donald Evans — 09/13/2022 Date

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3.0 INTRODUCTION, SCOPE AND APPLICABILITY

3.1 Introduction and Compliance References

Eurofins Edison's Quality Assurance (QA) Manual is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving the laboratory's QA Program. Governing SOPs are in place within the organization to ensure the proper execution of this QA Manual (refer to Appendix 1). This manual and referenced documents are required reading for all personnel.

The laboratory is a team of people who work together to serve the health and environmental needs of society through science and technology. We offer comprehensive expertise in environmental laboratory applications and client relations and maintain a national perspective in terms of quality.

As such, this QA Manual has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009 and 2016; ISO/IEC Guide 17025:2005 and 2017. Policies and procedures listed in Appendix 1 are compliant with the National Divisional Support Center (NDSC) Quality Management Plan (QMP) for Eurofins Environment Testing America and the various accreditation and certification programs which are held by the laboratory to support environmental work (Appendix 2). The QMP provides a summary of Eurofins Environment Testing America quality and data integrity system. It contains requirements and general guidelines under which all Eurofins Environment Testing America facilities shall conduct their operations.

Refer to Appendix 3 for a list of additional references for which this QA Manual is compliant; and Appendix 4 for the compliance crosswalk of this manual to the TNI & ISO/IEC Guide 17025 requirements.

3.2 Terms and Definitions

A QA Program is a system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the local management level through company goals, from guidance at the executive management level, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. Our program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization to better serve our clients.

Refer to Appendix 5 for a list of Terms and Acronyms.

3.3 Scope / Fields of Testing

The laboratory analyzes a broad range of environmental and industrial samples. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The QA Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The Program also contains guidelines on maintaining documentation of analytical processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found our active LIMS method database. Our areas of expertise include:

Standard Services	Specialty Services
• Volatiles • Semivolatiles • Metals • Pesticides/PCBs/Herbicides • Petroleum Hydrocarbons • Waste Characterization • Non-Potable Water Testing • Drinking Water Testing • Soil and Surface Water Testing	• 1,4-Dioxane • Free Cyanide • PCBs by EPA 680

NOTE: All current certificates and scopes of accreditation are available on the laboratory's website at https://www.eurofinsus.com/environment-testing.

The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory President, Laboratory Director/Business Unit Manager and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory President,, Laboratory Director/Business Unit Manager and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

3.4 Management of the Manual

3.4.1 Review Process

This manual is reviewed every two years by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the QMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control procedures (refer to SOP No. ED-GEN-002, *Document Control*).

4.0 MANAGEMENT REQUIREMENTS

4.1 Overview

The laboratory is a local operating unit of Eurofins Environment Testing America. The laboratory's operational and support staff have the day-to-day independent operational authority under the direction of the Laboratory President, Laboratory Director/Business Unit Manager and is supported by the NDSC QA team. The laboratory management staff includes directors,

managers and group leaders. The organizational chart of the management staff are presented in Figure 4-1. Individual departmental staff lists are maintained in the laboratory's internal intranet.

4.2 Roles and Responsibilities

In order for the QA Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The responsibility for quality resides with every employee of the laboratory. All employees have access to the QA Manual, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks impartially and in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

4.2.1 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)

The Vice President (VP) of QA/EHS reports directly to Eurofins Environment Testing America Chief Operating Officer (COO). With the aid of the NDSC Quality Team Members, Business Unit Managers and Laboratory Directors, the VP-QA/EHS has the responsibility for the establishment, general overview and maintenance of the Quality Assurance and EH&S Programs within Eurofins Environment Testing America. Additional responsibilities include:

- Review of QA/QC and EHS aspects of NDSC Official Documents, national projects and expansions or changes in services.
- Work with various organizations outside of the laboratory to further the development of quality standards and represent the laboratory at various trade meetings.
- Prepare monthly reports for quality and EH&S metrics across the environmental testing laboratories and a summary of any quality and EH&S related initiatives and issues.
- With the assistance of the Executive Management and the EHS Managers, maintenance and implementation of the Eurofins Environment Testing America Environmental, Health and Safety Program.

4.2.2 Quality Directors

There are four (4) Quality Directors within NDSC that report directly to the VP-QA/EHS. These Quality Directors have oversight of the general overview and maintenance of the QA Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Monitors laboratory internal audit findings;
- Identifies common laboratory weaknesses and monitors corrective action closures.
- Develops NDSC quality guidance documents and management tools for ensuring and improving compliance;
- Monitors and communicates DoD/DoE requirements;
- Monitors and communicates regulatory and certification requirements;
- Training and OnBoarding
- Laboratory assessments, mentoring, and interventions
- Track/drive root cause investigations and corrective action plans
- Builds knowledge base for preventive actions

4.2.3 Quality Information Manager

The Quality Information works directly with the NDSC Quality Directors and EHS Managers; and reports directly to the VP-QA/EHS. The Quality Information Manager is responsible for the management of:

- NDSC Official Documents
- TALS/LIMS Certification Module Data
- Company's Intranet website
- Company's Regulatory Limits Database
- Subcontract laboratory and approved vendor information
- Internal and External client support for various company groups (e.g., Client Services, EH&S, Legal, IT, Sales) for both quality and operational functions
- Communicate regulatory information and lists

4.2.4 Environmental Health and Safety (EH&S) Managers

There are 3 EH&S Managers within NDSC that report directly to the VP-QA/EHS. These EH&S Managers have oversight of the general overview and maintenance of the EH&S Program within the Eurofins Environment Testing America laboratories. Supported tasks include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for Eurofins Environment Testing America locations.
- Coordination/preparation of the Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

4.2.5 Ethics and Compliance Officers (ECOs)

The NDSC VP-QA/EHS and Corporate Counsel are designated Ethics and Compliance Officer (ECO). Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire executive management personnel and lab management staff.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the President, COO, Laboratory Director or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratory's regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

4.2.6 Laboratory Director

The Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their business unit President. The Laboratory Director is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Provides one or more technical managers for the appropriate fields of testing. If the Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Manager to temporarily perform this function. If the absence exceeds 35 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented. Works with Eurofins Environment Testing Human Resources for hiring of new personnel.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures company human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory. Assesses laboratory capacity and workload.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Contributes to the continuous improvement of the laboratory operations.
- Maintains an awareness of technical developments and regulatory requirements.
- Captains the management team, consisting of the QA Manager, the Technical Manager(s), and the Operations Manager as direct reports.

4.2.7 Quality Assurance (QA) Manager or Designee

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system at the laboratory where they work. The QA Manager is also responsible for any service centers connected with their laboratory that perform analytical tests, such as short holding time analyses for pH.

The QA Manager reports directly to the Laboratory Director and their NDSC Quality Director. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. The NDSC QA Team may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA office to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Have functions independent from laboratory operations for which he/she has quality assurance oversight.
- Have documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Have a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arrange for or conducting internal audits on quality systems and the technical operation
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QA Manual or laboratory SOPs shall be investigated following procedures outlined in Section 14 and if deemed necessary may be temporarily suspended during the investigation.
- Maintaining and updating the QA Manual.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Objectively monitor standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Performing technical data audits and method audits to ensure consistency and compliance with regulatory requirements.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or NDSC QA Team.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.

- Participate in the vendor and supplier approval process, including subcontractors.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Communication to the relevant regulatory authorities when there are management or facility changes that impact the laboratory.
- Ensuring communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025

4.2.8 Technical Manager or Designee

The Technical Manager(s) report(s) directly to the Operations Manager – Organic and Operations Manager – Inorganic. (Note: Technical Managers at the Edison lab serve a dual role as Department Supervisors. See Section 4.2.11). . He/she is accountable for all analyses and analysts under their experienced supervision and for compliance with the ISO 17025:2017 Standard. The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, and reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Providing training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.

- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinating sample management from “cradle to grave,” insuring that no time is lost in locating samples.
- Scheduling all QA/QC-related requirements for compliance, e.g., MDLs, etc.
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.
- Compliance with ISO 17025.

4.2.9 Operations Manager

The Operations Manager manages and directs the analytical production sections of the laboratory. The lab employs and Organics Operations Manager and an Inorganic Operations Manager. They report directly to the Laboratory Director and act as co-Technical Managers for their areas of responsibility. He/She assists the Technical Manager in determining the most efficient instrument utilization. More specifically, he/she:

- Evaluates the level of internal/external non-conformances for all departments
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Manager and QA Manager and in compliance with regulatory requirements.
- Works with the Preventive Maintenance Coordinator to ensure that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

4.2.10 Project Manager (PM)

Members of the laboratory Client Services/Project Management Group are responsible for organizing and managing client projects. Clients are assigned a project manager who serves as their primary contact at the laboratory. It is the PM’s responsibility to act as the client advocate by communicating client requirements to laboratory personnel and ensuring that clients provide complete information needed by the laboratory to meet those requirements – including all verbal communications.

- Scheduling sample submissions, sample container orders and sample pick-up via the laboratory courier service.
- Confirming certification status

- Coordinating and communicating turnaround time (TAT) requirements for high priority samples/projects.
- Answering common technical questions, facilitating problem resolution and coordinating technical details with the laboratory staff.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample status reports or results prior to receipt of the final report.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.

4.2.11 Department Supervisors

Department Supervisors (aka Department Managers) report to their respective Operations Managers (Organic or Inorganic). Department Supervisors in conjunction with their Operations Manager serve as Technical Managers (see Section 4.2.8 above) for their department. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 5.3), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.
- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Manager, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Manager, Operations Manager, and/or BU / Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.

- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

4.2.12 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Manager, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Manager, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

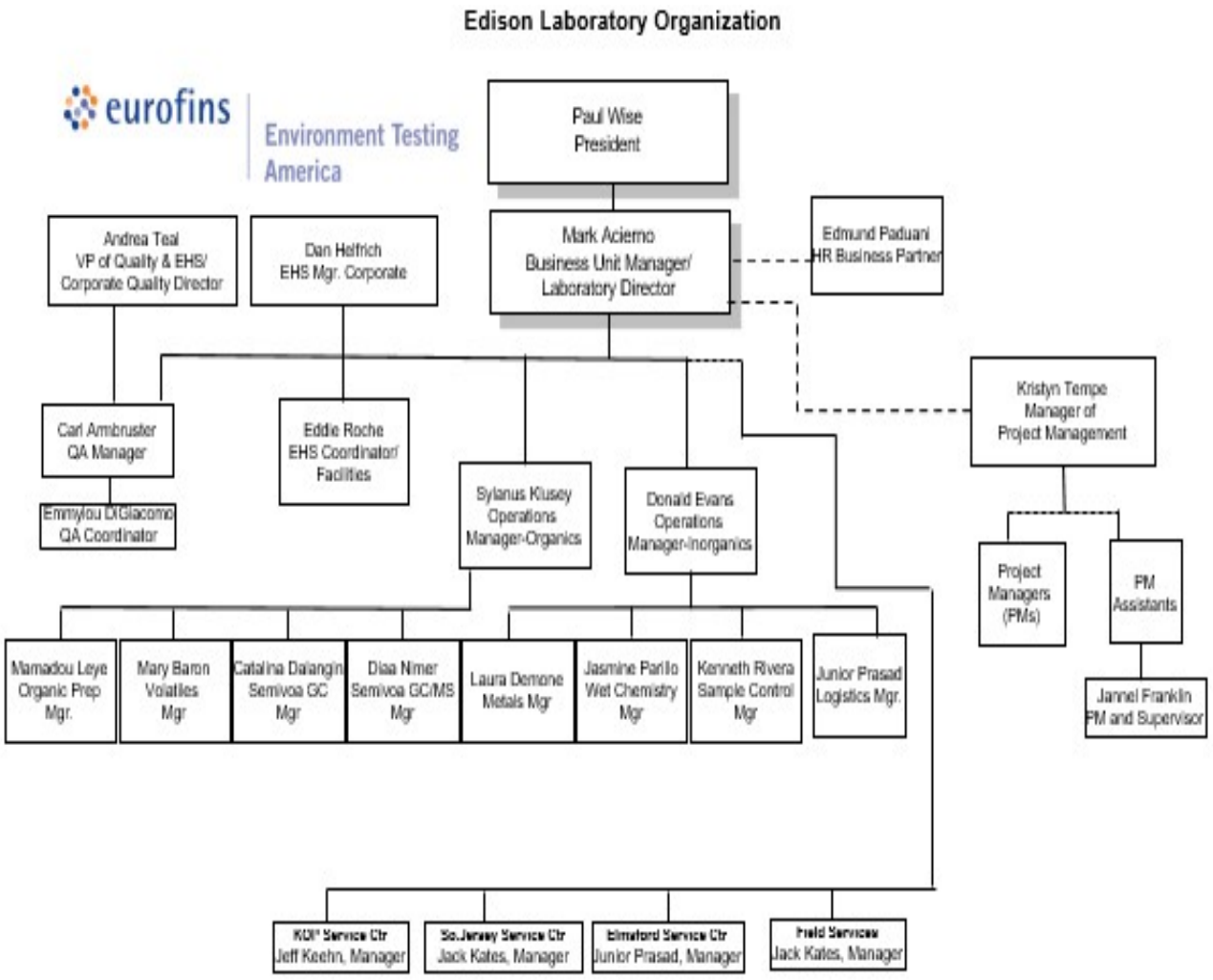
4.3 Business Continuity and Contingency Plans

Various policies and practices are in place to address continuity of business and contingency plans to ensure continued operations or minimal disruption in operations should unplanned events (natural disasters, unexpected management changes, etc.) occur. Deputies are identified for all key management personnel. Deputies would temporarily fill a role if the primary is absent for more than 15 consecutive calendar days. The deputies must meet the same qualifications as the primary person should they be required to take on the responsibilities. The QA Manager communicates to the relevant regulatory authorities when there are management or facility changes that impact the laboratory. Changes in the technical director must be communicated within a period of time and in the manner dictated by each regulatory authority.

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Mark Acierno Laboratory Director	In the event of absence the Laboratory Director's responsibilities are shared by the Laboratory Operations Manager, the Quality Assurance Manager and the Client Services Manager, as appropriate
Carl Armbruster Quality Manager	Emmylou Digiacomo Quality Assurance Specialist Mark Acierno Laboratory Director
Sylvanus Klusey Organic Operations Manager	Donald Evans Inorganics Operations Manager
Donald Evans Inorganics Operations Manager	Sylvanus Klusey Organic Operations Manager
VOA, SVOA & Organic Prep Department/Technical Managers	Sylvanus Klusey Organic Technical Manager
Wet Chemistry, Metals Department/ Technical Managers	Donald Evans Inorganics Operations Manager
Dan Helfrich EH&S Manager	Edward Roche EH&S Coordinator
Kristyn Tempe Manager of Project Managers	Mark Acierno Laboratory Director
John Kates South Jersey/King of Prussia Service Center Manager	Alonzo Hall, Sample Control Tech. South Jersey Service Center Jeffery Keehn, Sample Control Tech. King of Prussia Service Center
John Kates Fields Services Manager	Stephen Schulze Thomas Lesinski Field Specialists – Edison
Junior Prasad Logistics Manager/Elmsford Service Center Manager	Donald Evans Inorganics Operations Manager

Figure 4-1. Laboratory Organization Chart



5.0 PERSONNEL

5.1 Overview

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

5.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for laboratory employees are outlined in job descriptions maintained by Eurofins Environment Testing America Human Resources.

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)

Specialty	Education	Experience
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Managers – **General**	Bachelor's Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Managers – **Wet Chem** only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

5.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics Statement – New Hires and Annually	1 week of hire & annually	All

Required Training	Time Frame	Employee Type
Ethics – New Hires and Annually	60 days of hire and annually	All
Data Integrity – New Hires and Annually	60 days of hire and annually	Technical and PMs
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status and records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics violations). This information is maintained in the employee's secured personnel file.

Evidence of successful training could include such items as:

- Adequate documentation of training within operational areas, including one-on-one technical training for individual technologies, and particularly for people cross-trained.
- Analysts knowledge to refer to QA Manual for quality issues.
- Analysts following SOPs, i.e., practice matches SOPs.
- Analysts regularly communicate to supervisors and QA if SOPs need revision, rather than waiting for auditors to find problems.

Further details of the laboratory's training program are described in the Laboratory Training SOP ED-GEN-022, *Training*.

5.4 Data Integrity and Ethics Training Program

The laboratory's Ethics and Data Integrity Program is discussed in Section 6.2. Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.

- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (855-910-0005) is maintained by the NDSC.

6.0 ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

6.1 Overview

The laboratory is a 42,000 ft^2 secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

6.2 Environment

Laboratory accommodation, test areas, energy sources, and lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

When the laboratory performs laboratory activities at sites or facilities outside its permanent control, it shall ensure that the requirements related to facilities and environmental conditions of this document are met.

Specific requirements for facility and environmental conditions, as well as periodic monitoring of conditions, are given in the Environmental Health & Safety Manual plus each laboratory's Facility Addendum.

6.3 Work Areas

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory. Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

6.4 Responding to Emergencies

Employees must be aware of procedures to respond to all emergencies that might occur in the workplace. Employees must be familiar with the location and proper operation of all emergency equipment, evacuation routes and designated assembly areas for all areas where they work. Refer to the NDSC EH&S Manual Document No. CW-E-M-001. Sec. 7 and the laboratory's local EH&S addendum for complete details. These documents provide direction for situations where

normal operations of the laboratory are not possible (e.g., electrical failures, heating/air conditioning failures, fire/building evacuation, computer failures, hazardous material spills, injury to employees, pandemic flu, disruption of phone service, etc.)

In the event that the building or information technology (IT) systems would be severely challenged, a designated disaster recovery team, which includes Facility Management, Maintenance, Safety, Laboratory/Executive Management, Public Relations, IT, QA and other applicable personnel depending on the scope of the disaster, would assemble at a designated area to assess the situation and formulate a plan.

6.5 Building Security

The laboratory is considered a secure facility. All outside doors (except the main lobby entrance) are locked during normal business hours to prevent unauthorized entry. (An attendant monitors this entrance at all times.) Building keys and alarm codes are distributed to employees as necessary.

All visitors to the laboratory must sign in and out in a visitor's logbook that is located in the lobby. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. Both visitors and vendors must review and sign specific EH&S forms; and are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

Signs are posted in the laboratory designating employee only areas - "Authorized employees beyond this point".

7.0 QUALITY SYSTEM

7.1 Quality Policy Statement

The Quality Policy statement gives employees clear requirements for the production of analytical data. As an organization, all personnel are committed to high quality professional practice, testing and data, and service to our clients.

We strive to provide the highest quality data achievable by:

- Reading and understanding all of the quality documents applicable to each position and implementing the process in our work.
- Following all recordkeeping requirements; describing clearly and accurately all activities performed; recording "real time" as the task is carried out; understanding that it is never acceptable to "back date" entries and should additional information be required at a later date, the actual date and by whom the notation is made must be documented.
- Ensuring data integrity through the completeness, consistency, impartiality and accuracy of the data generated. Data is attributable, legible, contemporaneously recorded, original or a true copy, and accurate (ALCOA). This applies to manual paper documentation and electronic records.
- Providing accountability and traceability for each sample analyzed through proper sample handling, labeling, preparation, instrument calibration/qualification/validation, analysis, and reporting; establishing an audit trail (the who, what, when, and why) that identifies date, time, analyst, instrument used, instrument conditions, quality control samples (where appropriate and/or required by the method), and associated standard material.

- Emphasizing a total quality management process which provides impartiality, accuracy, and strict compliance with agency regulations and client requirements, giving the highest degree of confidence; understanding that meeting the requirements of the next employee in the work flow process is just as important as meeting the needs of the external client.
- Providing thorough documentation and explanation to qualify reported data that may not meet all requirements and specifications, but is still of use to the client; understanding this occurs only after discussion with the client on the data limitations and acceptability of this approach.
- Responding immediately to indications of questionable data, out-of-specification occurrences, equipment malfunctions, and other types of laboratory problems, with investigation and applicable corrective action; documenting these activities completely, including the reasons for the decisions made.
- Providing a work environment that ensures accessibility to all levels of management and encourages questions and expression of concerns on quality issues to management. Eurofins recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff
- Continually improve systems and manage risk to support quality improvement efforts in laboratory, administrative and managerial activities

7.2 Ethics and Data Integrity

Eurofins Environment Testing America is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The laboratory operates our Ethics and Data Integrity program under the guidance of Eurofin's Key Guidance Document (KGD). The elements of our program include:

- An Ethics Policy (NDSC Document No. CW-L-P-004) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (NDSC Document No. CW-L-S-002).
- Procedures and guidance for recalling data if necessary (NDSC Document No. CW-Q-S-005).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 17).
- Produce results, which are accurate and include QA/QC information that meets client pre-defined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Provide procedures and guidance to ensure the impartiality and confidentiality of all data and customer information

- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

7.3 Quality System Documentation

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance (QA) Manual – Each laboratory has a lab-specific QA manual.
- NDSC Official Documents – Each laboratory may use the Guidance (instructional use) documents at their discretion. Template documents are process documents that the laboratory's need to implement locally by using the document as is or as an outline to define their internal practices that meet the minimum requirements of the template. Required documents need to be implemented as is and listed in the laboratory's document control list.
- Key Guidance Documents (KGDs) - Documents compiled at the Group Service Centre (GSC) level by Functional Leaders (document owners) aimed at providing specific Eurofins groups of employees with guidelines necessary for the good conduct of their respective work.
- Laboratory SOPs and Policies – General and Technical
- Work Instructions – A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).

7.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Quality Management Plan (QMP)
- NDSC Guidance Documents
- KGDs
- Laboratory Quality Assurance Manual (QA Manual)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

NOTE: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the QMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QA Manual shall take precedence over the QMP in those cases.

7.4 QA/QC Objectives for the Measurement of Data

Quality Assurance (QA) is responsible for developing planned activities whose purpose is to provide assurance to all levels of management that a quality program is in place within the laboratory, and that it is functioning in an effective manner that is consistent with the requirements of NELAP, ISO 17025, and any other regulatory agencies (i.e., states) in which the laboratory maintains accreditation.

Quality Control (QC) is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. The client is responsible for developing the QAPP; however, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS). Each laboratory SOP defines the required QC indicators.

7.4.1 Precision

The objective is to meet the performance for precision demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

7.4.2 Accuracy

The objective is to meet the performance for accuracy demonstrated for The objective is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet DQOs of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

7.4.3 Representativeness

The objective is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. Refer to laboratory SOPs for subsampling and homogenization techniques appropriate to the analytical method.

7.4.4 Comparability

The objective is to provide analytical data for which the accuracy, precision, representativeness, and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

Comparability is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision, and reporting limits with those of other laboratories.

7.4.5 Completeness

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope, or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

7.4.6 Selectivity

Selectivity is defined as the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), and specific electrodes (separation and identification).

7.4.7 Sensitivity

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (above the Method Detection Limit) or quantified (above the Reporting Limit).

7.5 Criteria for Quality Indicators

The laboratory maintains tables, housed in LIMS, that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated, and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in lab SOP ED-GEN-026, Evaluation of Analytical Accuracy and Precision Through The Use of Control Charts.

7.6 Statistical Quality Control

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs.. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts use the current limits entered into LIMS. The QA department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in lab SOP ED-GEN-026, Evaluation of Analytical Accuracy and Precision Through The Use of Control Charts and Section 26. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

7.6.1 QC Charts

The QA Manager may evaluate QC control charts as needed to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

7.7 Quality System Metrics

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 18). These metrics are used to drive continuous improvement in the laboratory's Quality System.

8.0 DOCUMENT CONTROL

8.1 Overview

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance (QA) Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- NDSC Documents[1]
- KGDs[1]

[1]Includes locally implemented documents that are document controlled within the laboratory's document control system. The NDSC and/or KGD documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving NDSC Official

Documents is found in Document No. CW-Q-S-001, NDSC Document Control and Archiving. KGD documents are controlled according to this document. The laboratory's internal document control procedure is defined in SOP No. ED-GEN-002, Document Control.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data, and final reports.

8.2 Document Approval and Issue

The pertinent elements of a document control system includes a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number, and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a responsible manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then provided to all applicable operational units. Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years and revised as appropriate. Changes to documents occur when a procedural change warrants.

8.3 Procedures for Document Control Policy

For changes to the QA Manual, SOPS and Work Instructions refer to SOP No. ED-GEN-002, Document Control. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder for the applicable revision.

8.4 Obsolete Documents

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. ED-GEN-002, Document Control.

9.0 SERVICE TO THE CLIENT

9.1 Overview

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily fit into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals, and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another Eurofins facility on the same LIMS or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 10 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or Eurofins Edison are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

9.2 Review Sequence and Key Personnel

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet

the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the Client Relationship Manager or Proposal Team, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in NDSC Document No. CA-L-P-002**,** Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Contract Administrator
- Laboratory Project Manager
- Laboratory Directors and/or Technical Managers
- Account Executives
- Quality Managers
- Laboratory Environmental Health and Safety Managers/Directors

The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The Sales Director, Contract Administrator, Account Executive or Proposal Coordinator then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

9.3 Balancing Laboratory Capacity and Workload

Evaluating laboratory capacity to perform specific projects is the responsibility of the Vice-President, Laboratory Directors and Managers, and the Client Services director and manager. Many analysts are cross-trained to perform a variety of tests, and there is redundant equipment available in case of malfunctions. This minimizes the need to evaluate small and medium size projects against capacity available to complete them. Large and complex projects are reviewed against capacity estimates before bids are submitted to ensure that the client's analysis schedule is met. Regularly scheduled meetings are held between laboratory management, PMs, Client Services and QA personnel to review progress with current projects, as well as special requirements of new work scheduled for the laboratory. Laboratory capacity and backlog is tracked on a continuous basis using information from the Laboratory Sample Information System (LIMS) including turnaround time, and work in-house..

9.4 Documentation

The Contracts Department maintains copies of all signed contracts**.** The Manager of Project Management maintains a copy locally.

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. This documentation is maintained by the laboratory PM on the lab's network server.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Account Executive. A copy of the contract and formal quote will be filed with the laboratory PM and the Laboratory Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client and a copy of all related email correspondence.

9.4.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, a PM is assigned to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements, They coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new project information to maximize production and client satisfaction, while maintaining quality. Project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

Any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document (e.g., letter, e-mail, variance, contract addendum), which has been signed by both parties.

Such changes are also communicated to the laboratory. The laboratory staff is introduced to the modified requirements via the PM or the individual laboratory Technical Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

9.5 Special Services

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all

client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 17 and 25).

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assisting client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

When the client requests a statement of conformity to a specification or standard based on the analysis performed by the laboratory (e.g., pass/fail, in-tolerance/out-of-tolerance), the decision rule shall be clearly defined. Unless inherent in the requested specification or standard, the decision rule selected shall be communicated to the client. Associated reporting requirements are addressed in Section 25.2.18.

9.6 Client Communication

PMs are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Managers are available to discuss any technical questions or concerns that the client may have.

9.7 Reporting

The laboratory works with our clients to produce any special communication reports required by the contract.

9.8 Client Feedback and Surveys

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. Eurofins Sales and Marketing teams periodically develop lab and client specific surveys to assess client satisfaction. Client satisfaction surveys are sent to the client via email with the final report deliverables for the client's job(s).

When a complaint is received, we determine, to the best of our ability, the extent of the issue and what data is in question. The person receiving the complaint documents this information and promptly forwards it to the appropriate management personnel where the work in question was performed. If a data reporting error is discovered, the final report and/or data must be regenerated with the correct value(s).

The PM is responsible for entering client concerns into an NCM in TALS LIMS. In some cases, an ICAT is initiated to address and document the situation. While an individual issue may not warrant a formal investigation, QA monitors these issues for potential trends and will issue an ICAT if a trend is evident.

10.0 SUBCONTRACTING OF TESTS

The laboratory may subcontract tests to other laboratories if the requested testing is not routinely performed in our laboratory. To a lesser extent, samples may need to be subcontracted to an overflow laboratory to ensure hold times and/or turn-around-times (TAT) are met.

Testing is only subcontracted with the client's knowledge and approval. The <define> must notify the client in writing when any of their requested analyses will be subcontracted to another lab. Client approval must be obtained in writing before samples are shipped.

Subcontract laboratories are selected based on their qualifications and accreditations. The subcontractor is requested to sign a Laboratory Analytical Services Subcontract. If projects require a specific agency certification (i.e. individual state agencies, NELAP, DoD, PALA, ISO 17025), only an appropriately accredited laboratory is used. The client may also have a list of laboratories to be used for subcontracting. In these cases, the evaluation of the subcontract laboratory is made by the client.

Data obtained from subcontract laboratories is clearly marked as such when reported by the laboratory. The data are submitted to the client in the format obtained from the subcontractor.

10.1 Overview

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the Eurofins Environment Testing America laboratories. The phrase "work sharing" refers to internal transfers of samples between the Eurofins Environment Testing America laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity, or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to the NDSC Documents on Subcontracting Procedures (CW-L-S-004) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in TNI/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-TNI accredited work where required.

PMs or other responsible Client Service members, for the Export Lab (i.e., the Eurofins Eurofins Environment Testing America laboratory that transfers samples to another laboratory) are responsible for obtaining client approval prior to subcontracting any samples. The laboratory will advise the client of a subcontract arrangement in writing and when possible approval from the client shall be obtained and retained in the project folder. Standard Eurofins Environment Testing America Terms & Conditions include the flexibility to subcontract samples within the Eurofins Environment Testing America laboratories. Therefore, additional advance notification to

clients for intra-laboratory subcontracting is not necessary unless specifically required by a client contract.

Note: In addition to the client, some regulating agencies (e.g., USDA) or contracts require notification prior to placing such work**.**

10.2 Qualifying and Monitoring Subcontractors

Whenever a PM becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- Subcontractors specified by the client - In these circumstances, the client assumes responsibility for the quality of the data generated from the use of a subcontractor.
- Subcontractors reviewed by Eurofins Environment Testing America – Firms which have been reviewed by the company and are known to meet standards for accreditations (e.g., State, TNI and DoD/DOE); technical specifications; legal and financial information.

A listing of vendors is available on the Eurofins Environment Testing America intranet site.

All Eurofins Environment Testing America laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations and can adhere to the project/program requirements. Client approval is not necessary unless specifically required by the contract. In these cases, the client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs as detailed in NDSC Document No. CA-C-S-001, Work Sharing Process.

10.2.1 When the potential subcontract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Client Relations Manager (CRM). The CRM requests that the PM begin the process of approving the subcontract laboratory. Refer to the NDSC Document No. CW-L-S-004, Subcontracting Procedures.

Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability and forwarded to the NDSC Quality Information Manager (QIM) for review. After the NDSC QIM reviews the documents for completeness, the information is forwarded to the Finance Department for formal signature and contracting with the laboratory. The approved vendor will be added to the approved subcontractor list on the intranet site, and the finance group is concurrently notified.

The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. Eurofins Environment Testing America does not certify laboratories. The subcontractors on our approved list can only be recommended to the extent that we would use them.

10.3 Oversight and Reporting

The status and performance of qualified subcontractors will be monitored by NDSC and includes an annual review process (ref.: NDSC Document No. SOP CW-L-S-004. Any problems identified will be brought to the attention of NDSC.

- Complaints shall be investigated. Documentation of the complaint, investigation, and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. Client Service personnel will notify all Eurofins Environment Testing America laboratories, NDSC and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Client Service Personnel, Laboratory Directors, QA Managers, and Sales Personnel.

Prior to initially sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented within the project records.

The laboratory's certifications can be viewed on the company's website: https://www.eurofinsus.com/environment-testing/

10.3.1 All subcontracted samples must be accompanied by a Eurofins Edison Chain of Custody (COC). A copy of the original COC sent by the client must be available in LIMS for all samples workshared within Eurofins Environment Testing America. Client COCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client COCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI accredited work must be identified in the subcontractor's report as appropriate. If TNI accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratory's EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

Note: The results submitted by a Eurofins Environment Testing America work sharing laboratory may be transferred electronically and the results reported by the Eurofins Environment Testing America sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

10.4 Contingency Planning

The full qualification of a subcontractor may be waived to meet emergency needs. This decision and justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and COC.

In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation

requirements will still be applicable, but the subcontractor need not have signed a subcontract agreement with Eurofins Environment Testing America at this time.

The use of any emergency subcontractor will require the PM to complete a New Vendor Add Form in order to process payment to the vendor and add them to LIMS. This form requires the user to define the subcontractor's category/s of testing and the reason for testing.

10.5 Use of NELAP and A2LA Logo

It is not laboratory policy to use these logos on any company letterhead, including analytical reports.

11.0 PURCHASING SERVICES AND SUPPLIES

11.1 Overview

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with NDSC Document No. CW-F-S-007, Eurofins Environment Testing America Fixed Asset Acquisition, Retention and Safeguarding

Contracts will be signed in accordance with NDSC Document No. CW-F-P-002, Company-Wide Authorization Matrix Policy. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in the NDSC Document No. CW-F-P-004, Guidance on Procurement and Contracts Policy. RFP's allow the laboratory to determine if a vendor is capable of meeting requirements such as supplying all of the laboratory facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

11.2 Glassware

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

11.3 Reagents, Standards & Supplies

Purchasing guidelines for equipment, consumables, and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with NDSC Document No. CA-Q-S-001, Solvent & Acid Lot Testing & Approval. Approval information for the solvents and acids tested under Document No. CA-Q-S-001 is stored on the laboratory's SharePoint site, under Solvent Approvals. A master list of all tested materials, as well as the certificates of analysis for the materials, is stored in the same location.

11.3.1 Purchasing

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst completes the Material Request Sheet when requesting reagents, standards, or supplies. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use.

The analyst must provide the master item number (from the master item list that has been approved by the Technical Manager), item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the Technical Manager prior to placing the order. The purchasing manager places the order.

11.3.2 Receiving

It is the responsibility of the analyst or department manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to document the date materials were received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. This is documented through the addition of the received date and initials to the information present on the daily order log.

The analyst or department manager verifies the lot numbers of received solvents and acids against the pre-approval lists. If a received material is listed as unapproved, or is not listed, it is sequestered and returned to the vendor. Alternatively, the laboratory may test the material for the intended use, and if it is acceptable, document the approval on the approval list. Records of any testing performed locally are maintained on the shared “public” folder on the computer network.

Materials may not be released for use in the laboratory until they have been inspected, verified as suitable for use, and the inspection/verification has been documented.

Safety Data Sheets (SDSs) are available online through the Company’s intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

11.3.3 Specifications

Methods used in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, analytical reagent grade will be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer’s expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturer's or SOP expiration date unless verified as outlined below.

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded. In this case, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison must show that the dry chemical/solvent meets CCV limits. Any such comparison studies will be maintained on-file and available for review in the QA Manager's office.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. To prevent a tank from going to dryness, or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of samples, standards or reagents must have a specific conductivity of less than 1-µmho/cm (or specific resistivity of greater than 1.0 megohm-cm) at 25ºC. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified clean by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Manager or QA Manager.

11.3.4 Storage

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Storage conditions are per the NDSC Environmental Health & Safety Manual, Document No. CW-E-M-001, the local laboratory EH&S manual addendum and method SOPs or manufacturer instructions.

11.4 Purchase of Equipment / Instruments / Software

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in NDSC Document No. CA-T-P-001 Qualified Products List are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

11.5 Services

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 22. The need for service is determined by analysts and/or Technical Managers. The service providers that perform the services are approved by the Technical Manager.

Analytical balances are serviced and calibrated annually in accordance with SOP No.ED-GEN-010, Calibration of Analytical Balances. The calibration and maintenance services are performed on-site, and the balances are returned to use immediately following successful calibration. Calibration certificates are filed for reference. If the calibration was unsuccessful, the balance is immediately removed from service and segregated pending either further maintenance or disposal.

Calibration services for support equipment such as thermometers, weight sets, autopipettors, etc., are obtained from vendors with current and valid ISO 17025 accreditation for calibration of the specific piece of equipment. Prior to utilizing the vendor's services, the vendor's accreditation status is verified. Once the equipment has been calibrated, the calibration certificates are reviewed by the QA department, and documentation of the review is filed with the calibration certificates. The equipment is then returned to service within the laboratory

11.6 Suppliers

The laboratory selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts).

This process is defined in the NDSC Document No. CW-F-P-004, Procurement & Contracts Policy. The level of control used in the selection process is dependent on the anticipated

spending amount and the potential impact on the laboratory's business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Purchasing Group by completing a Vendor Performance Report.

The Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

Suppliers are subject to re-evaluation, as deemed appropriate, through the use of Vendor Performance Reports used to summarize and review to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the purchasing system.

11.6.1 New Vendor Procedure

Laboratory employees who wish to request the addition of a new vendor must complete a Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with laboratory employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Services Director are consulted with vendor and product selection that have an impact on quality.

12.0 COMPLAINTS

12.1 Overview

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures client knowledge that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 14 (Corrective Actions) and is documented following SOP No. ED-GEN-003, Control of Non-Conformances and Corrective Action. A copy of this procedure will be made available to any interested party on request.

12.2 External Complaints

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according SOP No. ED-GEN-003, Control of Non-Conformances and Corrective Action.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and documenting complaints
- Acknowledging receipt of complaint, whenever possible
- Complaint investigation and service recovery
- Process improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

12.3 Internal Complaints

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 14. In addition, Executive Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 14.

12.4 Management Review

The number and nature of client complaints is reported by the QA Manager to the Laboratory Director and Quality Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Systems Review (Section 18).

13.0 CONTROL OF NON-CONFORMING WORK

13.1 Overview

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 14).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 14. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 21. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical Manager and QA Manager,, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non-TNI state would need to note the change made to how the method is normally run

13.2 Responsibilities and Authorities

Under certain circumstances, the Laboratory Director, a Technical Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate**.** Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, and the Technical Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an ECO (e.g., the VP-QA/EHS) and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, VP of for due cause as well as authorize the resumption of work.

13.3 Evaluation of Significance and Actions Taken

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

The NDSC Document entitled Data Recalls (CW-Q-S-005) is the procedure to be followed when it is discovered that erroneous or biased data may have been reported to clients or regulatory agencies.

The NDSC Document entitled Internal Investigations (CW-L-S-002) is the procedure to be followed for investigation and correction of situations involved alleged incidents of misconduct or violation of the company's ethics policy.

Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in NDSC Document No.CW-Q-S-005.

13.4 Prevention of Nonconforming Work

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

13.5 Method Suspension / Restriction (Stop Work Procedures)

In some cases, it may be necessary to suspend/restrict the use of a method or target analyte which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 13.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line. The QA Manager will also initiate a corrective action report as described in Section 14 if one has not already been started. A copy of any meeting notes and agreed upon steps should be e-mailed by the laboratory to their Business Unit President; VP-QA & EHS. This e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc.). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (e.g., Laboratory Director, Technical Manager, QA Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete.

14.0 CORRECTIVE ACTION

14.1 Overview

A major component of the laboratory's (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. The laboratory employs two systems to manage non-conformances. Issues suspected of being systematic in nature and for which root cause analysis and a formal Corrective Action Report (CAR) are documented in the Incident Corrective Action Tracking (ICAT) database. Routine batch non-conformances, events that are understood to be isolated in nature, are documented in the LIMS non-conformance memo (NCM) system.

14.2 General

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc.

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.
- Identify and track client complaints and provide resolution.

14.2.1 Non-Conformance Memo (NCM)

NCMs are used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits

- Isolated reporting / calculation errors
- Client complaints
- Discrepancies in materials / goods received vs. manufacturer packing slips (Forms of documentation other than NCMs in LIMS are also acceptable)

14.2.2 Corrective Actions Documented In the ICAT Database

- Internal and external audit findings
- Failed or unacceptable PT results
- Identified poor process or method performance trends
- Systematic reporting / calculation errors
- Data recall investigations
- Questionable trends that are found in the review of NCMs.
- Client complaints
- Excessive revised reports

The ICAT database is used to document background information, track the results of corrective action investigations and root cause analysis, and to provide reports of corrective action plans.

14.3 Closed Loop Corrective Action Process

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

14.3.1 Cause Analysis

- Upon discovery of a non-conformance event, the event must be defined and documented. An entry into the ICAT system must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical Manager, Laboratory Director, or QA Manager (or QA designee) is consulted.

14.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The ICAT record is used for this documentation.

14.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness. NDSC Document No. CA-Q-S-009, Root Cause Analysis, provides guidance on this procedure

Systematically analyze and document the root causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the root cause data from these incidents to identify root causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed and continue to plague the laboratory or operation.

14.3.4 Monitoring of the Corrective Actions

- The Technical Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Technical Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- The QA Manager reviews monthly NCM and ICAT records for trends. Highlights are included in the QA monthly report (refer to Section 18). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the NDSC Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

14.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 17.1.4, Special Audits.)

14.4 Technical Corrective Actions

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 13). The documentation of these procedures is through the use of an NCM or record in the ICAT system.

Table 14-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 14-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QA Manual Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the PM is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented**.**

14.5 Basic Corrections

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original uncorrected file must be maintained intact and a second corrected file is created. This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated. When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Table 14-1. Example – General Corrective Action Procedures

QC Activity ***(Individual Responsible for Initiation/Assessment)***	**Acceptance Criteria**	**Recommended Corrective Action**
Initial Instrument Blank *(Analyst)*	- Instrument response < MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc..
Initial Calibration Standards *(Analyst, Technical Manager(s))*	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Independent Calibration Verification (Second Source) *(Analyst, Technical Manager(s))*	- % Recovery within control limits.	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards *(Analyst, Data Reviewer)*	% Recovery within control limits.	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Matrix Spike / Matrix Spike Duplicate (MS/MSD) *(Analyst, Data Reviewer)*	- % Recovery within limits documented in TALS Method Limit Group	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.
Laboratory Control Sample (LCS) *(Analyst, Data Reviewer)*	- % Recovery within limits specified in TALS Method Limit Group	- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance. When not using marginal exceedances, the following exceptions apply: 1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes; 2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes. **Note:** If there is insufficient sample or the holding time cannot be met, contact client and report with flags.
Surrogates *(Analyst, Data Reviewer)*	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS. - Surrogate results outside criteria shall be reported with qualifiers.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Method Blank (MB) *(Analyst, Data Reviewer)*	< Reporting Limit [1]	- Reanalyze blank. - If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results. - Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is > 1/10 of the amount measured in the sample.
Proficiency Testing (PT) Samples *(QA Manager, Technical Manager(s))*	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits *(QA Manager, Technical Manager(s), Laboratory Director)*	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical Managers, QA Manager, NDSC-QA, Executive Management)	- NDSC Document No. CW-Q-S-005, Data Recall **or** local SOP No.	- Corrective action is determined by type of error. Follow the procedures in NDSC Document No. CW-L-S-002 **or** in lab SOP No. ED-GEN-003, Non-Conformances and Corrective Action.
Client Complaints *(Project Managers, Lab Director/Manager, Sales and Marketing)*	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 16 for an example) (QA Manager, Lab Director/Manager, *Technical Manager(s)*)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.

QC Activity *(Individual Responsible for Initiation/Assessment)*	Acceptance Criteria	Recommended Corrective Action
Health and Safety Violation (Safety Officer, Lab Director/Manager, *Technical Manager(s)*)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

Note:
1. Except as noted below for certain compounds, the method blank should be below the reporting limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit

15.0 PREVENTIVE ACTION / IMPROVEMENT

15.1 Overview

The laboratory's preventive action programs improve or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its QA Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, the laboratory continually strives to improve customer service and client satisfaction through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered through any of the following:

- review of the monthly QA Metrics Report,
- trending NCMs,
- review of control charts and QC results,
- trending proficiency testing (PT) results,
- performance of management system reviews,
- trending client complaints,
- review of processing operations, or
- staff observations.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. The metrics report is reviewed monthly by the laboratory management, NDSC QA Team, Local and Executive Management. These metrics are used ~~to~~ in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

Items identified as continuous improvement opportunities to the management system may be issued as goals from the annual management systems review, recommendations from internal audits, white papers, Lessons Learned, Technical Services audit report, Technical Best Practices, or as Executive or management initiatives.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action and non-conformances provides a valuable mechanism for identifying preventive action opportunities.

15.1.1 The following elements are part of a preventive action/process improvement system:

- Identification of an opportunity for preventive action or process improvement.
- Process for the preventive action or improvement.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action or improvement.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action or improvement.
- Close-Out by documenting any permanent changes to the Quality System as a result of the Preventive Action or Process Improvement. Documentation of Preventive Action/process Improvement is incorporated into the monthly QA reports, corrective action process and management review.

15.1.2 Any preventive actions/process improvement undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

16.0 CONTROL OF RECORDS

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. Exceptions for programs with longer retention requirements are discussed in Section 14.1.2.

16.1 Overview

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. More detailed information on retention of specific records is provided in NDSC

Document Nos. CW-L-P-001, Records Retention Policy, CW-L-WI-001,Records Retention/Storage Schedule and local SOP No. ED-GEN-024, Records Storage and Retention.>. Quality records are maintained by the QA department in a database, which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by department supervisors.

Table 16-1. Record Index

	Record Types [1]:	**Retention Time:**
Technical Records	- Raw Data - Logbooks[2] - Standards - Certificates - Analytical Records - MDLs/IDLs/DOCs - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals - Published Methods	Indefinitely
QA Records	- Certifications - Method and Software Validation / Verification Data	Indefinitely
QA Records	- Internal & External Audits/Responses - Corrective/Preventive Actions - Management Reviews - Data Investigation	5 Years from archival* **Data Investigation:** 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	- Sample Receipt & COC Documents - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
Administrative Records	Financial and Business Operations	Refer to NDSC Doc. No. CW-L-WI-001
	EH&S Manual, Permits	Indefinitely
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	Refer to HR Manual
	Administrative Policies	Indefinitely

	Record Types [1]:	Retention Time:
	Technical Training Records	7 years
	Legal Records	Indefinitely
	HR Records	Refer to NDSC Doc. No. CW-L-WI-001
	IT Records	Refer to NDSC Doc. No. CW-L-WI-001
	NDSC Governance Records	Refer to NDSC Doc. No. CW-L-WI-001
	Sales & Marketing	5 years
	Real Estate	Indefinitely

[1] Record Types encompass hardcopy and electronic records.
[2] Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
* Exceptions listed in Table 16-2.

16.1.1 All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss**.** All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Electronic data backups are performed as described in the Eurofins Data Back-Up Policy (CW-I-P-010, current revision). Backups occur at a frequency that ensures that no more than one business day of critical data will be lost in the event of a system failure. The Edison laboratory uses Citrix as part of its data management system. All laboratory data is copied offsite to the Eurofins Datacenter, which is located in Colorado. The Edison facility maintains a local instrument fileserver to which all local data is copied before it is transmitted to the Datacenter and a backup virtual server as redundancy. All instrument data files are located on the local file server, EDI0011 and each evening at 6:00pm ET entire instrument data folder structure is copied to the redundant virtual server EDICV01. Every evening starting at 10:00pm ET the data is then replicated from EDICV01 to the Datacenter in Colorado where it's backed up to tape.

Access to the data is limited to laboratory and company employees and shall be documented with an access log**.** Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for the full length of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as NDSC and/or KGD Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 16-2 have lengthier retention requirements and are subject to the requirements in Section 16.1.3.

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16.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 16-2 with their retention requirements. In these

cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Table 16-2. Example: Special Record Retention Requirements

Program	[1]Retention Requirement
Drinking Water – All States	10 years (lab reports and raw data) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
OSHA	30 years

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

16.2.1 The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.15.1 for more information.

16.2.2 The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.
- All information relating to the laboratory facilities' equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set). Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".

- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP No. ED-GEN-021, Data Review.
- Also refer to Section 19.15.1 'Computer and Electronic Data Related Requirements'.

16.3 Technical and Analytical Records

16.3.1 The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

16.3.2 Observations, data and calculations are recorded real-time and are identifiable to the specific task.

16.3.3 Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- Laboratory sample ID code;
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and

reporting conventions;

- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in LIMS and on specific analytical report formats.

16.3.4 All logbooks used during receipt, preparation, storage, analysis, and reporting of samples or monitoring of support equipment shall undergo a periodic, documented supervisory or peer review.

16.4 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

16.4.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

16.5 Administrative Records

The laboratory also maintains the administrative records in either electronic or hard copy form.

Refer to Table 16-1.

16.6 Records Management, Storage and Disposal

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access**.**

The laboratory has a record management system (a.k.a., document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in LIMS – no logbooks are used to record that data**.** Records are considered archived when noted as such in the records management system (a.k.a., document control).

16.6.1 Transfer of Ownership

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of NDSC. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

16.6.2 Records Disposal

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

17.0 AUDITS

17.1 Internal Audits

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to Executive Management..

Audits are conducted and documented as described in the NDSC Document No. on performing Internal Auditing, SOP No. CW-Q-S-003. The types and frequency of routine internal audits are described in Table 17-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Table 17-1. Types of Internal Audits and Frequency

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or NDSC QA	All areas of the laboratory annually
Method Audits QA Technical Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to NDSC CW-Q-S-003)	QA Technical Audits Frequency: 50% of methods annually
SOP Method Compliance	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to NDSC CW-Q-S-003)	SOP Compliance Review Frequency: • Every 2 years • Every 1 year (Drinking Water Methods)
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI-field of testing or as dictated by regulatory requirements

17.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, Eurofins Data Integrity and Ethics Policies (see Sec. 7.2), TNI quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed for effectiveness & sustainability.

The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

17.1.2 QA Technical Audits

QA technical audits assess data authenticity and analyst integrity. These audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, electronic audit miner programs (e.g., Chrom AuditMiner) are used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

17.1.3 SOP Method Compliance

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Manager or qualified designee at least every two years. It is also recommended that the work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

17.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

17.1.5 Performance Testing

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-potable Water and Soil.

It is Eurofins policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written investigations for unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

17.2 External Audits

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is Eurofins policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with

access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response. Audit responses are due in the time allotted by the client or agency performing the audit.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

17.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 and 2016 TNI standards.

17.3 Audit Findings

Audit findings are documented using the corrective action process and database (see Section 14). The laboratory's corrective action responses may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Technical Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

Clients must be notified promptly in writing of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24 hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

18.0 MANAGEMENT REVIEWS

18.1 Quality Assurance Report

The QA Department is responsible for preparing a comprehensive monthly metrics report to Management to keep them apprised of current quality issues. This report fosters

communication, review, and refinement of the QA system to evaluate the suitability of policies and procedures to meet both regulatory and laboratory quality objectives.

The NDSC QA team compiles information from all of the Environment Testing laboratories monthly metrics reports for the Executive Management team. This report includes notable information and concerns regarding the laboratories QA program and a listing of new regulations that may potentially impact the laboratories.

18.2 Annual Management Review

The Laboratory Management team (Laboratory Director, Technical Managers, QA Manager) conducts a review annually of its quality systems to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. NDSC personnel is be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to LIMS. The laboratory will summarize any critical findings that cannot be solved by the lab and report them to Corporate

This management systems review NDSC Document No. CW-Q-S-004, Work Instruction No. CW-Q-WI-003 and local SOP No. ED-GEN-004, Quality Management Review uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
    - Adequacy of staff, equipment and facility resources.
    - Adequacy of policies and procedures.
    - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.
- For labs analyzing radioactive samples, also include the following:
    - Radiation health and safety

- Radioactive hazardous waste management
- Radioactive materials management

- Evaluation of overall risk, including risks to impartiality, confidentiality, reporting statements of conformity, and nonconforming work.

A report is generated by the QA Manager and management. The report is distributed to the, President of the Business Unit, the Lab Director/Business Unit Manager, and Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

18.3 Potential Integrity Related Managerial Reviews

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The NDSC Document No. CW-L-S-002, Internal Investigations shall be followed. All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

The Eurofins Environment Testing America Presidents, Business Unit Managers, Laboratory Directors and NDSC Team are informed any current data integrity or data recall investigations.

19.0 TEST METHODS AND METHOD VALIDATION

19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

19.2 Standard Operating Procedures (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most

recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to NDSC Document No. CW-Q-S-002, Writing a Standard Operating Procedure.
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD/DOE SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

Note: If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the PM. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data. Refer to Appendix 6 for a list of the currently accepted U.S. EPA analytical method references used by the laboratory.

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

19.4.1.1 Client Supplied Methods

Most of the client-supplied method requirements presented to us involve achieving specific quality control criteria, limits of quantitation (LOQ), and/or method detection limits (MDL) using standard EPA methods. These requirements are communicated to the appropriate technical groups prior to the project start up. Each technical group evaluates the scope of work and the requirements to ensure the criteria can be met using the standard EPA method. The data is monitored to ensure the criteria are met throughout the project. The PM notifies the client if there is a more appropriate method available or if the client's criteria cannot be achieved on a certain sample matrix (i.e., due to matrix or dilutions).

Occasionally, we are asked to transfer a non-standardized method from a client into our lab or to develop a new method, when one is not available. In the case of a method transfer, we set up the client's method and perform some initial evaluation. After the initial evaluation, we may make recommendations on how to improve method performance. If the method appears to be adequate, we determine linearity, specificity, precision, accuracy, MDL, and LOQ by performing calibrations, analyzing method blanks, and carrying out method detection limit and IDOC studies.

In the case of method development, we work with the client and/or data user to determine the level of validation required ensuring that the method meets its intended purpose. In addition to the elements above, we also determine standard and sample stability and robustness depending on the scope of the project. Typically, a standard operating procedure is written and submitted to the client with the results of the validation. These steps are completed prior to analysis of field samples. Data related to the setup of the method are archived.

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19.4.1.2 Procedural Deviations

Analysts are required to follow a documented method for all tests performed; and any deviations from analytical methods must be documented, approved, and justified in an appropriate and consistent manner. We classify method deviations as either being a planned deviation or an unplanned deviation. In general, the following information is captured to document both types of situations:

- Description of the situation
- Reason or justification for the deviation
- Impact the deviation had on the testing

- Signature/date of analyst performing the test
- Signature/date of QA and Laboratory management approving the deviation
- Signature/date of client approval, if necessary

Deviations to written procedures are documented in raw data records or through the ICAT system. Both types of documentation require management and QA review and approval.

19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (DOC) (Lab SOP ED-GEN-022, Training) is performed whenever there is a change in instrument type (e.g., new instrumentation), matrix, method or personnel (e.g., analyst has not performed the test within the last 12 months).

Note: The laboratory shall have a DOC for all analytes included in the methods that the laboratory performs, and proficiency DOCs for each analyst shall include all analytes that the laboratory routinely performs. Addition of non-routine analytes does not require new DOCs for all analysts if those analysts are already qualified for routine analytes tested using identical chemistry and instrument conditions.

The initial demonstration of capability must be thoroughly documented and approved by the Technical Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (e.g., retention time window study).

Note: In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Note: IDOC procedures are further detailed in Lab SOP ED-GEN-022, Training.

The spiking standard used must be prepared independently from those used in instrument calibration.

The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

19.4.3.1 At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

19.4.3.2 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

19.4.3.3 When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

19.4.3.4 Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

19.4.3.5 When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

19.5 Laboratory Developed Methods and Non-Standard Methods

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

19.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

19.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

When changes are made to any validated methods, the influence of such changes shall be documented and, if appropriate, a new validation shall be performed.

19.6.1.1 **Determination of Method Selectivity** – Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

19.6.1.2 **Determination of Method Sensitivity** – Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Detection limit studies are conducted as described in Section 19.7. Where other protocols for estimations and/or demonstrations of sensitivity are required by regulation or client agreement, these shall be followed.

19.6.1.3 **Relationship of Limit of Detection (LOD) to the Limit of Quantitation (LOQ)** – An important characteristic of expression of sensitivity is the distinction between the LOD and the LOQ. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The LOQ is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias, equivalent to the laboratory's routine reporting limit (RL). For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the LOQ. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the LOQ, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

19.6.1.4 **Determination of Interferences** – A determination that the method is free from interferences in a blank matrix is performed.

19.6.1.5 **Determination of Range** – Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

19.6.1.6 **Determination of Accuracy and Precision** – Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of

reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

19.6.1.7 **Documentation of Method** – The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

19.6.1.8 **Continued Demonstration of Method Performance** – Continued demonstration of method performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

19.7 Method Detection Limit (MDL) / Limits of Detection (LOD)

The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017. The MDL is equivalent to the TNI LOD or DL, and is also equivalent to the DoD/DOE Quality Systems Manual (QSM) DL. The working or final MDL is the higher of the MDL value determined from spikes (MDLs) and the MDL value determined from blanks (MDLb). An initial MDL study shall be performed during the method validation process and when the method is altered in a way that can reasonably be expected to change its sensitivity. On-going data are collected during each quarter in which samples are being analyzed. At least once every 13 months the MDLs and MDLb are re-calculated and re-evaluated using data collected during the preceding period. Details of the laboratory's procedure for conducting MDL studies are given in NDSC Document No.CA-Q-S-006.

19.8 Verification of Detection Limits

If it is found during the re-evaluation of detection limit results that more than 5% of the spiked samples do not return positive numeric results that meet all method qualitative identification criteria, then then spiking level shall be increased and the initial MDL study pre-performed at the new spiking concentration..

19.9 Instrument Detection Limits (IDL)

The IDL is sometimes used to assess the reasonableness of the MDL or in some cases required by the analytical method or program requirements. IDLs are most commonly used in metals analyses but may be useful in demonstration of instrument performance in other areas.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

19.10 Limit of Quantitation

The LOQ shall be at a concentration equivalent to the lowest calibration standard concentration, with the exception of methods using a single-point calibration, and shall be greater than the MDL. The LOQ is verified by preparing and analyzing spikes at concentrations 1-2X the selected LOQ, employing the complete analytical process.

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit or by a DL check samples at or below the LOQ. The LOQ is verified annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements

19.11 Retention Time Windows

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specified in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept on-file and available for review. Complete details are available in the laboratory SOPs.

19.12 Evaluation of Selectivity

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

19.13 Estimation of Uncertainty of Measurement

19.13.1 Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty" defined as the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

19.13.2 Uncertainty is not error. Error is a single value (i.e., the difference between the true result and the measured result). On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

19.13.3 The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent

recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

19.13.4 To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent uncertainties at approximately the 99% confidence level with a coverage factor of k = 3. As an example, for a reported result of 1.0 mg/L with an LCS recovery range of 50 to 150%, the estimated uncertainty in the result would be 1.0 +/- 0.5 mg/L.

19.13.5 In the case where a well-recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

19.14 Sample Reanalysis Guidelines

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within $\pm$ 1 reporting limit for samples $\leq$ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.

- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor or Laboratory Director if unsure.

19.15 Control of Data

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

19.15.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below.

The laboratory is currently using the Eurofins LIMS system, which has been highly customized to meet the needs of the laboratory.

19.15.1.1 Maintain the Database Integrity – Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, documentation of system failures and corrective actions taken, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

19.15.1.2 Ensure Information Availability – Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

19.15.1.3 Maintain Confidentiality – Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

19.15.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged using NDSC Document No. CA-Q-S-002, Acceptable Manual Integration Practices.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

19.15.2.1 All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

19.15.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (µg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (µg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1**%.** Units are defined in each lab SOP.

19.15.2.3 In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

19.15.2.4 For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a formatter for significant figure criterion for each analyte.

19.15.2.5 The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS, electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

19.15.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 14.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Manager/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

19.15.4 Review / Verification Procedures

Review procedures are outlined in Lab SOP ED-GEN-021, Data Review to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also utilizes NDSC Document No. CA-Q-S-002, Acceptable Manual Integration Practices to ensure the authenticity of the data . The general review concepts are discussed below, more specific information can be found in the SOPs.

19.15.4.1 Log-In Review - The data review process starts at the sample receipt stage. Sample control personnel review chain-of-custody forms and project instructions from the project management group. This is the basis of the sample information and analytical instructions entered into the LIMS. The log-in instructions are reviewed by the personnel entering the information, and a second level review is conducted by the project management staff.

19.15.4.2 First Level Data Review - The next level of data review occurs with the analysts. As data are generated, analysts review their work to ensure that the results meet project and SOP requirements. First level reviews include inspection of all raw data (e.g., instrument output for

continuous analyzers, chromatograms, spectra, and manual integrations), evaluation of calibration/calibration verification data in the day's analytical run, evaluation of QC data, and reliability of sample results. The analyst transfers data into LIMS, data qualifiers are added as needed. All first level reviews are documented.

19.15.4.3 Second Level Data Review – All analytical data are subject to review by a second qualified analyst or supervisor. Second level reviews include inspection of all raw data (e.g., instrument output, chromatograms, and spectra) including 100% of data associated with any changes made by the primary analyst, such as manual integrations or reassignment of peaks to different analytes, or elimination of false negative analytes. The second review also includes evaluation of initial calibration/calibration verification data in the day's analytical run, evaluation of QC data, reliability of sample results, qualifiers and NCM narratives. Manual calculations are checked in second level review. All second level reviews are documented.

Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

19.15.4.4 Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

19.15.4.5 The results are then entered or directly transferred into the computer database and a .pdf is printed for the client.

19.15.4.6 As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that the COC is followed, cover letters / narratives are present, flags are appropriate, and project specific requirements are met. The Project Manager may also evaluate the validity of results for different test methods given expected chemical relationships.

19.15.4.7 Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

19.15.4.8 A brief overview of sample flow and information through the laboratory, as well as data review and validation, is presented in Figure 19-1.

19.15.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using NDSC Document No. CA-Q-S-002, Acceptable Manual Integration Practices.

19.15.5.1 The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

19.15.5.2 Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principles and policy and is grounds for immediate termination.

19.15.5.3 Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.

19.15.5.4 All manual integrations receive a second level review. Manual integrations must be indicated on ~~an~~ expanded scale “before” and “after” chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale “before” chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented NDSC approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

Figure 19-1. Example – Work Flow

Action	Personnel Involved
Bottle orders generated upon request - Bottles packed and shipped to the client under CoC	- Client Service Personnel / PM - Bottle Preparation Personnel
Samples received at laboratory - Unpacked and reconciled against client paperwork or CoC - Samples acceptance documented against completed (CoC)[1]	Sample Login Personnel
Samples are entered into LIMS - Job # assigned - Methods/Analytes linked from project - Sample labels generated - LIMS backlogs updated immediately	Sample Login Personnel

Action	Personnel Involved
Sample Storage - Short TATs and hold-time samples are delivered directly to the laboratory for analysis - Samples stored in assigned locations (refrigerated walk-in coolers, freezers, VOA refrigerators; etc.)	Sample Login Personnel
Sample Receipt Acknowledgement sent to client (when requested)	Sample Login Personnel
Sample Acquisition/Testing - Analysts collect samples from assigned storage. - Legal CoC samples are signed in-and-out of storage - Remaining sample returned to storage.	Analysts / Login Personnel
Preparation and analysis is performed according to selected analytical method and applicable Project notes - Raw data recorded/reviewed[2] - Data imported to LIMS	Analysts
LIMS performs calculations: - Data reviewed by primary/secondary analyst - Data approved as final	Data Process (LIMS / Analyst)
Generation/review/release of reports (automated through LIMS) - Electronic copy saved in LIMS	Project Management Assistants / Reporting Group
Electronic Data Deliverables (EDDs) generated (as applicable)	EDD Group
Hard copy and electronic batch raw data is archived according to data archiving procedures.	Analysts / IT

[1]Refer to Sec. 23.3.
[2]Analyses requiring the analyst's interpretation may involve manual data reduction before entry into LIMS.

20.0 EQUIPMENT and CALIBRATIONS

20.1 Overview

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of available laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

20.2 Preventive Maintenance

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Scheduled routine maintenance is defined in each method SOP. It is the responsibility of each Technical Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.
- Each entry in the instrument log includes the analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.
- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

If an instrument requires repair (subjected to overloading or mishandling), gives suspect results, or otherwise has shown to be defective or outside of specified limits it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back-up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

At a minimum, if an instrument is sent out for service or transferred to another facility, it must be recalibrated and the laboratory MDL verified (using an MDLV) prior to return to lab operations.

20.3 Support Equipment

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains “calibration only” ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file as detailed in lab SOP ED-GEN-010, Calibration of Analytical Balances.

20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to ± 0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters used in the laboratory are capable or measuring conductivity with an error not exceeding 1% or one umhos/cm, whichever is greater. The meters are also calibrated before each use with a known standard.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH, Conductivity, and Turbidity SOPs for further information.

20.3.3 Thermometers

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer.

- If the temperature measuring device is used over a range of 10°C or less, then a single point verification within the range of use is acceptable;
- If the temperature measuring device is used over a range of greater than 10°C, then the verification must bracket the range of use.

IR thermometers, digital probes and thermocouples are calibrated quarterly. IR Thermometers should be calibrated over the full range of use, including ambient, iced (4°C) and frozen (0°C to -5°C), per the Drinking Water Manual.

The digital NIST thermometer is recalibrated every five years (unless the thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the lab SOP ED-GEN-014, Thermometer Calibration..

20.3.4 Refrigerators/Freezer Units, Water baths, Ovens and Incubators

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, water baths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0ºC and ≤ 6 ºC.

Specific temperature settings/ranges for other refrigerators, ovens, water baths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for measurements, a label shall be applied to the device stating that it is not calibrated. Any device not regularly verified cannot be used for any quantitative measurements. Lab SOP ED-GEN-011, Calibration and Use of Lab Pipettes, contains detailed information on this topic.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an “Accuracy and Precision Statement of Conformance” from Hamilton attesting established accuracy.

20.3.6 Autoclaves

Section reserved. Autoclaves are not currently in use at Eurofins Edison.

20.3.7 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated monthly by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 10%, the procedure is repeated and if the result is still greater than 10%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

20.4 Instrument Calibrations

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, and type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Each method that requires a standard curve must have that curve evaluated for Relative Standard Error as detailed in Eurofins document NDSC-QA-QP44940, Calibration Curves and the Selection of Calibration Points. The procedure for evaluating Relative Standard Error is documented in each applicable SOP.

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 14).

Note: Instruments are calibrated initially and as needed after that and at least annually (the annual requirement does not apply to Isotope dilution).

20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exceptions to these rules. ICP and ICPMS methods which define the working range with periodic linear dynamic range studies, rather than through the range of concentrations of daily calibration standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst at a different time or a different preparation would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

20.4.1.1 Calibration Verification

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 and 2016 TNI Standard. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification (ICV) is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications (CCV) may use the same source standards as the calibration curve.

Note: The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 and 2016 TNI Std. EL-V1M4 Sec. 1.7.2.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

Note: If an internal standard calibration is being used then bracketing calibration verification standards may not be required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the calibrations must be verified by an ICV analyzed immediately following initial calibration and before sample analysis. The ICV may be used as the first bracketing CCV, if criteria for both are met.

A continuing instrument calibration verification (CCV) is generally analyzed at the beginning of each 12-hour analytical shift during which samples are analyzed. The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12-hours of the beginning of the shift. For methods that have quantitation by external calibration models, a CCV is analyzed at the end of each analytical sequence. Some methods have more frequent CCV requirements. See specific SOPs. Most inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (e.g., GCMS) then bracketing standards are not required, only daily verifications are needed, except as specified by program or method requirements.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective action shall be performed. Once corrective actions have been completed and documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable and **reported based upon discussion and approval of the client** under the following special conditions:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported case narrative comment explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

20.4.1.2 Verification of Linear and Non-Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Additional details are provided in NDSC Document No. CA-Q-QM-001, Tentatively Identified Compounds (TICS) - GCMS Analysis.

Note: If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

20.6 GC/MS Tuning

Prior to any GC/MS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spectrometer, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally do not need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 20-1

Eurofins Edison– Instrumentation by Type

(see EDS-WI-002, Eurofins Edison Equipment List for full current listing)

GC	GC/MS	ICP	ICP-MS	Hg Analyzer	Auto Analyzer	IC	TOC	Spectrophoto meter
x	x	X	x	x	x	x	x	x

21.0 MEASUREMENT TRACEABILITY

21.1 Overview

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware and glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

21.2 NIST-Traceable Weights and Thermometers

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), or another accreditation organization that is a signatory to a MRA (Mutual Recognition Arrangement) of one or more of the following cooperations – ILAC (International Laboratory Accreditation Cooperation) or APLAC (Asia–Pacific Laboratory Accreditation Cooperation). A calibration certificate and scope of accreditation is kept on file at the laboratory.

21.3 Reference Standards / Materials

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared reference standards, to the extent available, are purchased from vendors that are accredited to ISO Guide 34 and ISO/IEC Guide 17025. All reference standards from commercial vendors shall be accompanied with a certificate that includes at least the following information:

- Manufacturer
- Analytes or parameters calibrated
- Identification or lot number
- Calibration method
- Concentration with associated uncertainties
- Purity

If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number

and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the true value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the–Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory's Environmental Health and Safety Manual.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company-wide purchase. [Refer to NDSC Document No. (CA-Q-S-001) Solvent and Acid Lot Testing and Approval]

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained [in the TALS Reagent Module. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material. Blended gas standard cylinders use a nominal concentration if the certified value is within +/-15%, otherwise the certified values is used for the canister concentration.

21.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

21.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (from LIMS)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained in the TALs Reagent Module.

21.4.3 In addition, the following information may be helpful:

- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and preparation/analytical batch records.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

22.0 SAMPLING

22.1 Overview

The laboratory provides sampling services. Sampling procedures are described in the following SOPs:

- ED-FLD-008 (Low Flow Well Purging & Sampling)
- ED-FLD-009 (Volume Average Purging & Sampling)
- ED-FLD-014 (Wastewater Sampling)

22.2 Sampling Containers

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Certificates of cleanliness for bottles and preservatives are provided by the supplier and are maintained at the laboratory. Alternatively, the certificates may be maintained by the supplier and available to the laboratory on-line.

22.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

22.3 Definition of Holding Time

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in days (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in hours (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. Holding times for

analysis include any necessary reanalysis. However, there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

22.4 Sampling Containers, Preservation Requirements, Holding Times

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. If method required holding times or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or “ASAP” is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

22.5 Sample Aliquots / Subsampling

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory’s responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots and subsampling are located in lab SOP ED-GEN-007, Subsampling.

23.0 HANDLING OF SAMPLES

It is the responsibility of the client to send us representative and/or homogeneous and properly preserved samples of the system from which they are drawn. The laboratory assumes that all multiple sample containers with the same designator/description and bottle type contain a homogeneous, representative sample.

The laboratory provides the appropriate sample containers, required preservative, chain-of-custody (COC) forms, shipping containers, labels, and custody seals. The laboratory also provides trip blanks and analyte-free water for field blanks. Preparation of methanol containers for field preservation of volatile soil samples is available.

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

23.1 Chain of Custody (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory’s custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect.

23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible. This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

When the sampling personnel deliver the samples directly to the laboratory personnel, the samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory personnel. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a laboratory courier. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by the laboratory when personnel at the fixed laboratory facility have physical contact with the samples.

Note: Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in log-in by date; it lists all receipts each date

23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

23.2 Sample Receipt

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are detailed in the laboratory's SOP No. ED-SPM-001, *Procedure For Sample Receipt, Login, Identification, And Storage* and summarized in the following sections.

23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented in a TALS Sample Receiving NCM and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

Sample Receiving personnel check and document preservation of non-volatile liquid samples after the samples have been entered into the LIMS and before they are released to the laboratory for testing or placed into storage. Any checks of volatile samples and samples for oil and grease are performed and documented at the time of analysis.

23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-1) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined.

23.3.1 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

23.3.2 Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into LIMS according to SOP No. ED-SPM-001, *Procedure For Sample Receipt, Login, Identification, And Storage*.

23.4 Sample Storage

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators, freezers or protected locations suitable for the sample matrix. Samples for metals analysis are stored unrefrigerated. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator or shelf (for metals samples) from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. All samples are kept in the refrigerators for 30 days after delivery of the final report to the client, which meets or exceeds most sample holding times. After 30 days the samples are disposed of or, upon client request moved to a sample archive area where they are stored for an additional time period agreed upon with the client or dictated by the applicable analytical program..

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of Eurofins Edison.

23.5 Hazardous Samples and Foreign Soils

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only.

Procedures for the handling and storage of hazardous samples are addressed in the Eurofins Safety Manual (Document No. CW-E-M-001) and in Eurofins Edison SOP No. ED-SPM-001 (*Sample Receipt, Login, Identification, and Storage*).

Procedures for the acceptance and handling of USDA regulated domestic and foreign soils are detailed in Eurofins Edison SOP No. ED-SPM-006 (*Procedure for Acceptance and Handling of Regulated Domestic and Foreign Soils).*

23.6 Sample Shipping

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a Eurofins Edison courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The laboratory's Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will not analyze the trip blanks that were supplied. However, in the interest of good client service, the laboratory will advise the client at the time of sample receipt that it was noted that they did not request analysis of the trip blank; and that the laboratory is providing the notification to verify that they are not inadvertently omitting a key part of regulatory compliance testing.

23.7 Sample Disposal

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures). All procedures in the laboratory's Environmental Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.

Figure 23-1. Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

Per State and/or Federal Regulation, the client is responsible to ensure that samples are shipped in accordance with DOT/IATA requirements, and that radioactive materials may only be delivered to licensed facilities. Any samples containing (or suspected to contain) Source, Byproduct, or Special Nuclear Material as defined by 10 CFR should be delivered directly to facilities licensed to handle such radioactive material. Natural material or ores containing naturally occurring radionuclides may be delivered to any Eurofins facility or courier as long as the activity concentration of the material does not exceed 270 pCi/g alpha or 2700 pCi/g beta (49 CFR Part 173).

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
    - *Client name, address, phone number and fax number (if available)*
    - *Project name and/or number*
    - *The sample identification*
    - *Date, time and location of sampling*
    - *The collectors name*
    - *The matrix description*
    - *The container description*
    - *The total number of each type of container*
    - *Preservatives used*
    - *Analysis requested*
    - *Requested turnaround time (TAT)*
    - *Any special instructions*
    - *Purchase Order number or billing information (e.g. quote number) if available*
    - *The date and time that each person received or relinquished the sample(s), including their signed name.*
    - *The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.*
    - Information must be legible
2) Samples must be properly labeled.
    - Use durable labels (labels provided by Eurofins are preferred)
    - Include a unique identification number
    - Include sampling date and time & sampler ID
    - Include preservative used.
    - Use indelible ink
    - Information must be legible

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.

4) Samples must be preserved according to the requirements of the requested analytical method

5) Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C and above freezing (0°C). For methods with other temperature criteria (e.g. some bacteriological methods require ≤ 10 °C), the samples must arrive within ± 2° C of the required temperature or within the method specified range.

5i.) Samples that are delivered to the laboratory on the same day they are collected may not meet the requirements of Section 5. In these cases, the samples shall be considered acceptable if the samples were received on ice.

5ii.) If sample analysis is begun within fifteen (15) minutes of collection, thermal preservation is not required.

5iii.) Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen (15) minutes of collection.

- Chemical preservation (pH) will be verified prior to analysis and documented, either in sample control or at the analyst's level. The project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.
- For Volatile Organic analyses in drinking water (Methods 502.2 or 524.2). Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
  - 1. Test for residual chlorine in the field prior to sampling.
    - If no chlorine is present, the samples are to be preserved using HCl as usual.
    - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCl.
  - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.
- **FOR WATER SAMPLES TESTED FOR CYANIDE (by Standard Methods or EPA 335)**
  - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH.
    - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered and qualify the results in the final report.
  - It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.
  - The laboratory must test the sample for oxidizing agents (e.g. Chlorine) prior to analysis and treat according to the methods prior to distillation. (ascorbic acid or sodium arsenite are the preferred choice).

Sample Holding Times

- Eurofins Edison will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time for us to ensure analysis.
- Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, Eurofins Edison will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted on the "Short Hold Time Detail Report" in the final report. Samples analyzed in the laboratory will be qualified on the final report with an 'H' to indicate holding time exceedance.

6) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. Eurofins Edison will supply a blank with the bottle order.

7) The project manager will be notified if any sample is received in damaged condition. Eurofins Edison will request that a sample be resubmitted for analysis.

8) Recommendations for packing samples for shipment.
   - Pack samples in Ice rather than "Blue" ice packs.
   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
   - Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
   - Fill extra cooler space with bubble wrap.

24.0 ASSURING THE QUALITY OF TEST RESULTS

24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. Quality control samples are to be treated in the exact same manner as the associated field samples being tested. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide

a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

24.3 Negative Controls

Table 24-1. Example – Negative Controls

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank [1]	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan). Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks [1]	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks [1]	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

[1] When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

24.4 Positive Controls

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.

If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, Toxaphene and PCBs in Method 608.3), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (see below) shall be used to control the test method. The

selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

24.5 Sample Matrix Controls

Table 24-3. Sample Matrix Control

Control Type	Details	
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency [1]	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency [1]	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates[2]	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency [1]	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency [1]	All organic and ICP methods as required by the analytical method.

Table 24-3. Sample Matrix Control

Control Type	Details	
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

[1] See the specific analytical SOP for type and frequency of sample matrix control samples.

[2] LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

24.6 Acceptance Criteria (Control Limits)

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

Note: For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated % Recovery acceptance (control) limits are generally established by taking $\pm$ 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).
- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.
- The maximum acceptable recovery limit will be 150%.
- The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

- If either the high or low end of the control limit changes by ≤ 5% from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

24.6.1 The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. Reference Eurofins Edison SOP No. ED-GEN-026 (Evaluation of Analytical Accuracy and Precision Through The Use of Control Charts).

24.6.2 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

24.6.3 If a surrogate standard falls outside the acceptance limits, and if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

24.7 Additional Procedures to Assure Quality Control

The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 17).

A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

- Use of formulae to reduce data is discussed in the method SOPs and in Section 20.
- Selection of appropriate reagents and standards is included in Section 11 and 21.
- A discussion on selectivity of the test is included in Section 7.
- Constant and consistent test conditions are discussed in Section 6.
- The laboratories sample acceptance policy is included in Section 23.

25.0 REPORTING RESULTS

25.1 Overview

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 9.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client. Review of reported data is included in Section 19.

25.2 Test Reports

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

25.2.1 A report title (e.g., Analytical Report)

25.2.2 The cover page shall include the laboratory name, address and telephone number.

25.2.3 A unique identification of the report (e.g., Eurofins Edison Job ID #) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

Note: Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

25.2.4 A copy of the chain of custody (COC).

- Any COCs involved with Subcontracting are included.

25.2.5 The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

25.2.7 Description and unambiguous identification of the tested sample(s) including the client identification code.

25.2.8 Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

25.2.9 Date reported or date of revision, if applicable.

25.2.10 Method of analysis including method code (EPA, Standard Methods, etc.).

25.2.11 Reporting limit.

25.2.12 Method detection limits (if requested)

25.2.13 Definition of Data qualifiers and reporting acronyms (e.g. ND).

25.2.14 Sample results.

25.2.15 QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

25.2.16 Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

25.2.17 A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.

25.2.18 A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory, except when information is provided by the client. When data is provided by the client there shall be a clear identification of it, and a disclaimer shall be put in the report when the client supplied data can affect the validity of the test.

25.2.19 A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

25.2.20 A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Authorized signatories are qualified Project Managers appointed by the Manager of Project Managers.

25.2.21 When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.

25.2.22 The laboratory includes a cover letter.

25.2.23 Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

25.2.24 When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

25.2.25 Appropriate laboratory certification number for the state of origin of the sample, if applicable.

25.2.26 If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, or how your lab identifies it). A complete report must be sent once all of the work has been completed.

25.2.27 Any non-Eurofins subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All Eurofins subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

25.2.28 A Certification Summary Report, where required, will document that, unless otherwise noted, all analytes tested and reported by the laboratory were covered by the noted certifications.

Note: Refer to the NDSC Document No. CA-I-P-002, Electronic Reporting and Signature Policy. for details on internally applying electronic signatures of approval.

25.2.29 Where the laboratory is responsible for the sampling stage, in addition to the requirements listed above, reports containing the results of sampling shall include the following, where necessary for the interpretation of test results:

- the date of sampling;
- unambiguous identification of the material sampled;
- the location of sampling plan and procedures, and deviations, addition to or exclusions from the sample procedures;
- a reference to the sampling plan and procedure, and deviations, additions to or exclusions from the sample procedures;
- details of any environmental conditions during sampling that affect the interpretation of test results;
- information required to evaluate measurement uncertainty for subsequent testing

25.3 Reporting Level or Report Type

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level 1 is a report with all of the elements outlined in Section 25.2 above, excluding 25.2.15 (QC data).
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in EDD (see below). Initial reports may be provided to clients by facsimile. Procedures used to ensure client confidentiality are outlined in Section 25.6.

25.4 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of Eurofins Edison services in addition to the test report as described in Section 25.2. When NELAP accreditation is required and both a test report and EDD are provided to the client, the official version of the test report will be the combined information of the report and the EDD. Eurofins Edison offers a variety of EDD formats including NJ Hazsite Deliverables, Excel, Dbase, GISKEY, and Text Files

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without

errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

25.5 Supplemental Information for Test

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as estimated.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

When, as requested by the client and agreed to by Eurofins Edison the report includes a statement of conformity to specification or standard (see Special Services, Section 7.4), the report shall clearly identify:

- to which results the statement applies,
- which specifications, standard or parts thereof are met or not, and
- the decision rule that was applied (unless the decision rule is inherent in the requested specification or standard, taking into account the level of risk (such as false accept and false reject and statistical assumptions) associated with the decision rule.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

Note: Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

25.6 Environmental Testing Obtained From Subcontractors

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the NDSC Document No. CW-L-S-004, Subcontracting.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of Eurofins are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

25.7 Client Confidentiality

The laboratory will ensure the highest standards of quality and integrity of the data and services provided to our clients

The laboratory is responsible for maintaining in confidence all client information obtained or created. In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

The laboratory will not intentionally divulge to any person (other than the client or any other person designated by the client in writing) any information regarding the services provided by the laboratory or any information disclosed to the laboratory by the client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

Information about the client obtained from sources other than the client (e.g., complainant, regulators) shall be confidential between client and the laboratory. The source of this information shall be confidential to the laboratory and shall not be shared with the client, unless agreed by the source.

Note: This shall not apply to the extent that the information is required to be disclosed by the laboratory under the compulsion of legal process. The laboratory will, to the extent feasible, provide reasonable notice to the client before disclosing the information..

Note: Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

25.7.1 Report deliverable formats are discussed with each new client. If a client requests that reports be e-mailed, the reports are to meet all requirements of this document.

25.8 Format of Reports

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

25.9 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "Rev (n)" where 'n' is the revision number. The revised report will have the words "Revision (n)" on the report cover page beneath the report date. Additionally, a section entitled "Revised Report" will appear on the Case Narrative page. A brief explanation of the reasons for the re-issue will be included in this section.

When the report is re-issued, a notation of "report re-issue" is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the re-issue and a reference back to the last final report generated. *For Example*: *Report was revised on 3/8/20 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 1/27/20 at 10:47am.*

25.10 Policies on Client Requests for Amendments

25.10.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely *no possible* impact on the interpretation of the analytical results and there is *no possibility* of the change being interpreted as misrepresentation by anyone inside or outside of our company.

25.10.2 Multiple Reports

The laboratory does not issue multiple reports for the same work order where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

Appendix 1. List of Governing Documents applicable to the QA Manual

NDSC Doc. No.	Title
CA-C-S-001	Work Sharing Process
CA-I-P-002	Electronic Reporting and Signature Policy
CA-L-P-002	Contract Compliance Policy
CA-Q-M-002	Quality Management Plan
CA-Q-QM-001	Guidance on Tentatively Identified Compounds (TICs) – GC/MS Analysis
CA-Q-S-001	Acid and Solvent-Lot Testing and Approval Program
CA-Q-S-002	Manual Integrations
CA-Q-S-006	Detection and Quantitation Limits
CA-Q-S-009	Root Cause Analysis
CA-T-P-001	Qualified Products List
CW-E-M-001	Environmental Health & Safety Manual
CW-F-P-002	Company-Wide Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy

CW-F-S-007	Fixed Asset Acquisition, Retention and Safeguarding
CW-I-M-001	IT Change Control Procedure Manual
CW-I-P-001	Data Back-up Policy
CW-L-P-001	Records Retention Policy
CW-L-P-004	Ethics Policy
CW-L-S-002	Internal Investigation
CW-L-S-004	Subcontracting
CW-Q-S-001	Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-Q-S-003	Internal Auditing
CW-Q-S-004	Management Systems Review
CW-Q-S-005	Data Recall Process
CW-Q-S-001	Document Control and Archiving
NDSC-QA-QP44940	Calibration Curves and the Selection of Calibration Points.
Local Laboratory Document No.	**Title**
ED-GEN-002	Document Control
ED-GEN-022	Training
ED-GEN-010	Calibration of Analytical Balances
ED-GEN-003	Control of Non-Conformances and Corrective Action
ED-GEN-024	Records Storage and Retention
ED-GEN-021	Data Review
ED-GEN-026	Evaluation of Analytical Accuracy and Precision Through The Use of Control Charts
ED-GEN-004	Quality Management Review
ED-GEN-014	Calibration of Laboratory Thermometers and Measurement of Temperatures in the Laboratory
ED-GEN-011	Calibration, Use, and Care of Laboratory Pipettes
ED-GEN-007	Subsampling
ED-FLD-008	Low Flow Monitoring Well Purging and Sampling
ED-FLD-009	Volume-Average Purging and Sampling
ED-FLD-014	Wastewater Sampling
ED-SPM-001	Procedure For Sample Receipt, Login, Identification, And Storage
ED-SPM-006	Procedure for Acceptance and Handling of Regulated Domestic and Foreign Soil
ED-SPM-008	Laboratory Waste Disposal Procedures

Appendix 2. List of Laboratory Certifications, Accreditations, Validations

The laboratory maintains accreditations, certifications, and approvals with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/ certification/licensing with the following organizations:

eurofins | Environment Testing America

Certification List

Laboratory	Program	Authority	Identification	Expiration Date
Edison	NELAP	New Jersey	12028	06/30/2023
Edison	NELAP	New York	11452	04/01/2023
Edison	NELAP	Pennsylvania	68-00522	02/28/2023
Edison	State	Connecticut	PH-0200	09/30/2022
Edison	State	DE Haz. Subst. Cleanup Act (HSCA)	N/A	01/01/2023
Edison	State	Massachusetts	M-NJ312	06/30/2023
Edison	State	Rhode Island	LAO00376	12/31/2022
Edison	US Federal Programs	USDA	P330-20-00244	11/03/2023

Appendix 3. References used to prepare the QA Manual

The QAM has been prepared to be consistent with the requirements of the following documents:

- ANSI/ASQC, E4-1994, "Specifications and Guidelines for Quality Management Systems for Environmental Data Collection and Environmental Technology Programs" (American National Standard, January 5, 1995, or most recent version)
- "EPA Requirements for Quality Management Programs" (QA/R-2) (EPA/240/B-01/002, May 31, 2006).
- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water,* EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, *Handbook for Analytical Quality Control in Water and Wastewater Laboratories,* EPA, March 1979.
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005) (DW labs only)*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- APHA, *Standard Methods for* the Examination of Water and Wastewater, 18th Edition, 19th, 20th, 21st, 22nd and on-line Editions.
- *U.S. Department of Energy* Order 414.1B, *Quality Assurance*, Approved April 29, 2004.
- *U.S. Department of Energy* Order 414.1C, *Quality Assurance*, June 17, 2005.
- *U.S. Department of Energy Order 414.1D, Quality Assurance, April, 25, 2011.*
- Nuclear Regulatory Commission (NRC) Quality Assurance Requirements.
- Marine Protection, Research, and Sanctuaries Act (MPRSA).
- Toxic Substances Control Act (TSCA).

Appendix 4. QA Manual Crosswalk with TNI and ISO/IEC 17025 Standards

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
-	QUALITY ASSURANCE MANUAL COVER PAGE	V1M2 Sec. 4.2.8.3		
1.0	Title Page			
2.0	Table of Contents	V1M2 Secs. 4.2.8.3-4.2.8.4		8.1.2, 8.2.1
3.0	Introduction, Scope and Applicability	V1M2 Sec. 4.2.8.4		
3.1	Introduction and Compliance References	V1M2 Secs. 1.1; 1.2; 2.0; 3.2; 4.1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.4; 8.3.1
3.2	Terms and Definitions	V1M2 Secs. 3.0; 4.2.4	4.2.4	
3.3	Scope /Field of Testing	V1M2 Secs. 1.2; 4.2.4	4.1.2; 4.2.4	5.3; 5.4; 8.2.1; 8.2.4
3.4	Management of The Manual	V1M2 Secs. 4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	4.2.1; 4.2.7; 4.3.3.2; 4.3.3.3	5.3
4.0	Management and Responsibilities	V1M2 Sec. 4		8.2.4; 8.2.5
4.1	Overview	V1M2 Secs. 4.1.1, 4.1.3; 4.1.5	4.1.1; 4.1.3; 4.1.5; 4.2.6	5.1; 5.2; 5.5; 5.6 ; 6.2.1; 6.2.4
4.2	Roles and Responsibilities	V1M2 Secs. 4.1.4; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.3; 4.1.5; 4.1.6; 4.2.1; 4.2.6; 5.2.4	4.1.1 to 4.1.3; 4.1.5; 5.5; 5.6; 6.2.1; 6.2.4; 6.2.6 8.2.2;
4.3	Business Continuity and Contingency Plan *(Prev. Deputies)*	V1M2 Secs. 4.1.5; 4.1.7.2; 4.2.7	4.1.5; 4.2.7	
5.0	PERSONNEL	V1M2 Secs. 5.2; 5.2.1	5.2.1	6.1; 6.2.3
5.1	Overview	V1M2 Secs. 5.2.2; 5.2.3; 5.2.5	5.2.2; 5.2.3; 5.2.5	6.2.2
5.2	Education and Experience Requirements for Technical Staff	V1M2 Secs. 5.2.1; 5.2.3; 5.2.4	5.2.1; 5.2.3; 5.2.4	6.2.2 to 6.2.4
5.3	Training	V1M2 Sec. 5.2.5	5.2.5	4.2.1; 6.2.2; 6.2.4; 6.2.5
5.4	Data Integrity and Ethics Training Program	V1M2 Sec. 4.2.8.1; 5.2.7		4.1.1
6.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS	V1M2 Sec. 5.3		6.1; 6.3.1
6.1	Overview	V1M2 Secs. 5.3.1; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.3; 5.3.4; 5.3.5	6.3.1
6.2	Environment	V1M2 Secs. 5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	5.3.1; 5.3.2; 5.3.3; 5.3.4; 5.3.5	6.3.1 to 6.3.5
6.3	Work Area	V1M2 Secs. 5.3.3; 5.3.4; 5.3.5	5.3.3; 5.3.4; 5.3.5	6.3.1
6.4	Responding to Emergencies			
6.5	Building Security	V1M2 Sec. 5.3.4	5.3.4	6.3.4
7.0	QUALITY SYSTEM			6.1; 8.2.4
7.1	Quality Policy	V1M2 Secs. 4.1.5; 4.2.2; 4.2.3; 4.2.8.3	4.1.5; 4.2.2; 4.2.3	8.2.3; 8.6.1
7.2	Ethics and Data Integrity	V1M2 Secs. 4.1.5; 4.16; 4.2.2; 4.2.8.1; 5.2.7	4.1.5; 4.2.2	4.1.1 to 4.1.3; 4.2.1; 6.2.1; 8.2.2; 8.2.3
7.3	Quality System Documentation	V1M2 Secs. 4.1.5; 4.2.2; 4.2.5	4.2.2; 4.2.5	8.2.4

Sec. No.	Title	2009 and 2016 TNI Standard Reference	ISO/IEC 17025:2005(E) Reference	ISO/IEC 17025:2017(E) Reference
7.4	QA/QC Objectives for the Measurement of Data	V1M2 Sec. 4.2.2	4.1.5; 4.2.2	6.2.4
7.5	Criteria for Quality Indicators			
7.6	Statistical Quality Control			
7.7	Quality System Metrics			
8.0	Document Control	V1M2 Secs. 4.2.7; 4.3.1; 4.3.2.2 ; 4.3.3.3; 4.3.3.4	4.2.7; 4.3.1; 4.3.2.2; 4.3.3.3; 4.3.3.4	8.2.4; 8.3.1
8.1	Overview			8.2.5; 8.3.1; 8.3.2
8.2	Document Approval and Issue	V1M2 Secs. 4.3.2; 4.3.2.1-4.3.2.3; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.2.3; 4.3.3.1	8.2.5; 8.3.2
8.3	Procedures for Document Control Policy	V1M2 Secs. 4.3.2.1–4.3.2.2; 4.3.3.1	4.3.2.1; 4.3.2.2; 4.3.3.1	8.2.5; 8.3.2
8.4	Obsolete Documents	V1M2 Secs. 4.3.2.1–4.3.2.2	4.3.2.1; 4.3.2.2	8.2.5; 8.3.2
9.0	SERVICE TO THE CLIENT	V1M2 Secs. 4.4.1 - 4.4.4	4.4.1; 4.4.2; 4.4.3; 4.4.4	7.1.1; 7.1.1.4; 7.1.1.5; 7.1.1.8; 7.1.2.1
9.1	Overview	V1M2 Secs. 4.4.5; 4.5.5; 5.7.1	4.4.5; 5.7.1	
9.2	Review Sequence and Key Personnel	V1M2 Sec. 4.4.5	4.4.5	7.1.1.6
9.3	Balancing Laboratory Work Load and Capacity			
9.4	Documentation	V1M2 Sec. 5.7.1	5.7.1	
9.5	Special Services	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.3; 7.1.1.7
9.6	Client Communication	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7
9.7	Reporting	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7
9.8	Client Feedback and Surveys	V1M2 Secs. 4.7.1-4.7.2	4.7.1; 4.7.2	7.1.1.7; 8.6.2
10.0	SUBCONTRACTING OF TESTS	V1M2 Secs. 4.4.3; 4.5.4	4.4.3; 4.5.4	
10.1	Overview	V1M2 Secs. 4.5.1 - 4.5.3; 4.5.5; 5.3.1	4.5.1; 4.5.2; 4.5.3; 5.3.1	6.6.1; 7.1.2.1; 7.1.2.2
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Appendix 5. Terms/Glossary and Acronyms (EL-V1M2 Sec. 3.1)

Terms/Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Anomaly: A condition or event, other than a deficiency, that may affect the quality of the data, whether in the laboratory's control or not.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Curve: The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Standard: A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM): A reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody (COC) Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified.

Confidential Business Information (CBI): Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. TNI and its representatives agree to safeguard identified CBI and to maintain all information identified as such in full confidentiality.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to Second Column Confirmation; Alternate wavelength; Derivatization; Mass spectral interpretation; Alternative detectors or Additional Cleanup procedures. (TNI)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Correction: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis**.** No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Integrity: The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements. (TNI)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item (ASQC), whether in the laboratory's control or not.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Field Blank: Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Holding Times: The maximum time that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

Internal Standard Calibration: Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Instrument Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is ± 100%. The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Least Squares Regression (1st Order Curve): The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

Limit(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: The MDL is the minimum measured quantity of a substance that can be reported with 99% confidence that the concentration is distinguishable from method blank results, consistent with 40CFR Part 136 Appendix B, August, 2017.

Limit(s) of Quantitation (LOQ) [a.k.a., Reporting Limit]: The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

(QS) Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts.

Drinking Water: Any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Air & Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (TNI)

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: See Limit of Detection (LOD)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Observation: A record of phenomena that (1) may assist in evaluation of the sample data; (2) may be of importance to the project manager and/or the client, and yet not at the time of the observation have any known effect on quality.

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI)

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within specified acceptance criteria. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item or service is of the type of quality needed and expected by the client. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (TNI)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC activities. (TNI)

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Second Order Polynomial Curve (Quadratic): The 2nd order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2nd order regression will generate a coefficient of determination (COD or r^2) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.99.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Spike: A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

Uncertainty: A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

Acronyms:

CAR – Corrective Action Report

CCV – Continuing Calibration Verification

CF – Calibration Factor

CFR – Code of Federal Regulations

COC – Chain of Custody

DOC – Demonstration of Capability

DQO – Data Quality Objectives

DUP - Duplicate

EHS – Environment, Health and Safety

EPA – Environmental Protection Agency

GC - Gas Chromatography

GC/MS - Gas Chromatography/Mass Spectrometry

HPLC - High Performance Liquid Chromatography

ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy

ICP/MS – ICP/Mass Spectrometry

ICV – Initial Calibration Verification

IDL – Instrument Detection Limit

IH – Industrial Hygiene

IS – Internal Standard
LCS – Laboratory Control Sample
LCSD – Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
LOD – Limit of Detection
LOQ – Limit of Quantitation
MDL – Method Detection Limit
MDLCK – MDL Check Standard
MDLV – MDL Verification Check Standard
MRL – Method Reporting Limit Check Standard
MS – Matrix Spike
MSD – Matrix Spike Duplicate
SDS - Safety Data Sheet
NELAP - National Environmental Laboratory Accreditation Program
PT – Performance Testing
TNI – The NELAC Institute
QAM – Quality Assurance Manual
QA/QC – Quality Assurance / Quality Control
QAPP – Quality Assurance Project Plan
RF – Response Factor
RPD – Relative Percent Difference
RSD – Relative Standard Deviation
SD – Standard Deviation
SOP – Standard Operating Procedure
TAT – Turn-Around-Time
VOA – Volatiles
VOC – Volatile Organic Compound

Appendix 6. Analytical Method References

Reference methods include:

- *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures; 40CFR Part 136 as amended by Method Update Rule; August 28, 2017.*
- *Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.*
- *Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.*
- *Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.*
- *Methods for the Determination of Organic Compounds in Drinking Water, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)*
- *Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994*
- *Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.*
- *Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th/ on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.*
- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008; Final Update V, August 2015.*
- *Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.*
- *Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)*
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL *NY Lab Id No: 10670*
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Dissolved Gases

Acetylene	RSK-175
Ethane	RSK-175
Ethene (Ethylene)	RSK-175
Methane	RSK-175
Propane	RSK-175

Fuel Additives

Methyl tert-butyl ether	EPA 524.2
Naphthalene	EPA 524.2

Metals I

Arsenic, Total	EPA 200.8 Rev. 5.4
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Cadmium, Total	EPA 200.8 Rev. 5.4
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Iron, Total	EPA 200.7 Rev. 4.4
Lead, Total	EPA 200.8 Rev. 5.4
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Mercury, Total	EPA 245.1 Rev. 3.0
Selenium, Total	EPA 200.8 Rev. 5.4
Silver, Total	EPA 200.7 Rev. 4.4
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4

Serial No.: 68522

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Metals II

Aluminum, Total	EPA 200.8 Rev. 5.4
Antimony, Total	EPA 200.8 Rev. 5.4
Beryllium, Total	EPA 200.8 Rev. 5.4
Nickel, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Thallium, Total	EPA 200.8 Rev. 5.4
Vanadium, Total	EPA 200.7 Rev. 4.4

Metals III

Calcium, Total	EPA 200.7 Rev. 4.4
Magnesium, Total	EPA 200.7 Rev. 4.4
Potassium, Total	EPA 200.7 Rev. 4.4
Sodium, Total	EPA 200.7 Rev. 4.4

Methylcarbamate Pesticides

3-Hydroxy Carbofuran	EPA 531.1
Aldicarb	EPA 531.1
Aldicarb Sulfone	EPA 531.1
Aldicarb Sulfoxide	EPA 531.1
Carbaryl	EPA 531.1
Carbofuran	EPA 531.1
Methomyl	EPA 531.1
Oxamyl	EPA 531.1

Miscellaneous

2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 1613B
Methyl iodide	EPA 524.2
Organic Carbon, Total	SM 21-23 5310C (-00)
Surfactant (MBAS)	SM 21-23 5540C (-00)

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Miscellaneous

Turbidity	SM 21-23 2130 B (-01)
	EPA 180.1 Rev. 2.0

Non-Metals

Alkalinity	SM 21-23 2320B (-97)
Calcium Hardness	SM 18-22 2340C (-97)
	SM 18-22 2340B (-97)
Chloride	EPA 300.0 Rev. 2.1
Color	SM 21-23 2120B (-01)
Cyanide	EPA 335.4 Rev. 1.0
Fluoride, Total	EPA 300.0 Rev. 2.1
	SM 21-23 4500-F C (-97)
Nitrate (as N)	EPA 353.2 Rev. 2.0
	EPA 300.0 Rev. 2.1
Nitrite (as N)	EPA 353.2 Rev. 2.0
	EPA 300.0 Rev. 2.1
Orthophosphate (as P)	SM 19, 21-23 4500-P E (-99)
Silica, Total	SM 21-23 4500-SiO2 C (-97)
Solids, Total Dissolved	SM 21-23 2540C (-97)
Specific Conductance	SM 21-23 2510B (-97)
Sulfate (as SO4)	EPA 300.0 Rev. 2.1

Perfluorinated Alkyl Acids

11CL-PF3OUDS	EPA 533
	EPA 537.1
4:2FTS	EPA 533
6:2FTS	EPA 533
8:2FTS	EPA 533

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Perfluorinated Alkyl Acids

9CL-PF3ONS	EPA 533
	EPA 537.1
ADONA	EPA 533
	EPA 537.1
Hexafluoropropylene Oxide Dimer Acic	EPA 533
	EPA 537.1
NETFOSAA	EPA 537.1
NMEFOSAA	EPA 537.1
Nonafluoro-3,6-Dioxaheptanoic Acid	EPA 533
Perflourotridecanoic Acid (PFTRDA)	EPA 537.1
Perfluordecanoic Acid (PFDA)	EPA 533
	EPA 537.1
Perfluoro-3-Methoxypropanoic Acid	EPA 533
Perfluoro-4-Methoxybutanoic Acid	EPA 533
Perfluorobutanesulfonic Acid (PFBS)	EPA 533
	EPA 537.1
Perfluorobutanoic Acid (PFBA)	EPA 533
Perfluorododecanoic Acid (PFDOA)	EPA 533
	EPA 537.1
Perfluoroheptanesulfonic Acid (PFHPS	EPA 533
Perfluoroheptanoic Acid (PFHPA)	EPA 533
	EPA 537.1
Perfluorohexanesulfonic Acid (PFHXS	EPA 533
	EPA 537.1
Perfluorohexanoic Acid (PFHXA)	EPA 533
	EPA 537.1
Perfluorononanoic Acid (PFNA)	EPA 533

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Perfluorinated Alkyl Acids

Perfluorononanoic Acid (PFNA)	EPA 537.1
Perfluorooctanesulfonic Acid (PFOS)	EPA 533
	EPA 537.1
Perfluorooctanoic Acid (PFOA)	EPA 533
	EPA 537.1
Perfluoropentanesulfonic Acid (PFPES	EPA 533
Perfluoropentanoic Acid (PFPEA)	EPA 533
Perfluorotetradecanoic Acid (PFTA)	EPA 537.1
Perfluoroundecanoic Acid (PFUNA)	EPA 533
	EPA 537.1
PFEESA	EPA 533

Trihalomethanes

Bromodichloromethane	EPA 524.2
Bromoform	EPA 524.2
Chloroform	EPA 524.2
Dibromochloromethane	EPA 524.2
Total Trihalomethanes	EPA 524.2

Volatile Aromatics

1,2,3-Trichlorobenzene	EPA 524.2
1,2,4-Trichlorobenzene	EPA 524.2
1,2,4-Trimethylbenzene	EPA 524.2
1,2-Dichlorobenzene	EPA 524.2
1,3,5-Trimethylbenzene	EPA 524.2
1,3-Dichlorobenzene	EPA 524.2
1,4-Dichlorobenzene	EPA 524.2
2-Chlorotoluene	EPA 524.2

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Volatile Aromatics

4-Chlorotoluene	EPA 524.2
Benzene	EPA 524.2
Bromobenzene	EPA 524.2
Chlorobenzene	EPA 524.2
Ethyl benzene	EPA 524.2
Hexachlorobutadiene	EPA 524.2
Isopropylbenzene	EPA 524.2
n-Butylbenzene	EPA 524.2
n-Propylbenzene	EPA 524.2
p-Isopropyltoluene (P-Cymene)	EPA 524.2
sec-Butylbenzene	EPA 524.2
Styrene	EPA 524.2
tert-Butylbenzene	EPA 524.2
Toluene	EPA 524.2
Total Xylenes	EPA 524.2

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2
1,1-Dichloropropene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2
1,2-Dichloroethane	EPA 524.2
1,2-Dichloropropane	EPA 524.2
1,3-Dichloropropane	EPA 524.2

Serial No.: 68522

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

NY Lab Id No: 10670

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Volatile Halocarbons

2,2-Dichloropropane	EPA 524.2
Bromochloromethane	EPA 524.2
Bromomethane	EPA 524.2
Carbon tetrachloride	EPA 524.2
Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2
Methylene chloride	EPA 524.2
Tetrachloroethene	EPA 524.2
trans-1,2-Dichloroethene	EPA 524.2
trans-1,3-Dichloropropene	EPA 524.2
Trichloroethene	EPA 524.2
Trichlorofluoromethane	EPA 524.2
Vinyl chloride	EPA 524.2

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All approved analytes are listed below:

Acrylates

Acrolein (Propenal)	EPA 8260D
	EPA 624.1
Acrylonitrile	EPA 8260D
	EPA 624.1
Ethyl methacrylate	EPA 8260D
Methyl acrylonitrile	EPA 8260D
Methyl methacrylate	EPA 8260D

Amines

1,2-Diphenylhydrazine	EPA 625.1
	EPA 8270E
1-Naphthylamine	EPA 8270E
2,3-Dichloroaniline	EPA 625.1
2-Naphthylamine	EPA 8270E
2-Nitroaniline	EPA 8270E
3-Nitroaniline	EPA 8270E
4,4'-Methylenebis(2-chloroaniline)	EPA 8270E
4-Chloroaniline	EPA 8270E
4-Nitroaniline	EPA 8270E
5-Nitro-o-toluidine	EPA 8270E
Aniline	EPA 625.1
	EPA 8270E
Carbazole	EPA 625.1
	EPA 8270E
Diphenylamine	EPA 8270E
Pronamide	EPA 8270E
Propionitrile	EPA 8260D
Pyridine	EPA 625.1

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Amines

Pyridine	EPA 8270E

Benzidines

3,3'-Dichlorobenzidine	EPA 625.1
	EPA 8270E
3,3'-Dimethylbenzidine	EPA 8270E
Benzidine	EPA 625.1
	EPA 8270E

Chlorinated Hydrocarbon Pesticides

4,4'-DDD	EPA 8081B
	EPA 608.3
4,4'-DDE	EPA 8081B
	EPA 608.3
4,4'-DDT	EPA 8081B
	EPA 608.3
Aldrin	EPA 8081B
	EPA 608.3
alpha-BHC	EPA 8081B
	EPA 608.3
alpha-Chlordane	EPA 8081B
	EPA 608.3
beta-BHC	EPA 8081B
	EPA 608.3
Chlordane Total	EPA 8081B
	EPA 608.3
Chlorobenzilate	EPA 8270E
delta-BHC	EPA 8081B

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Chlorinated Hydrocarbon Pesticides

delta-BHC	EPA 608.3
Diallate	EPA 8270E
Dieldrin	EPA 8081B
	EPA 608.3
Endosulfan I	EPA 8081B
	EPA 608.3
Endosulfan II	EPA 8081B
	EPA 608.3
Endosulfan sulfate	EPA 8081B
	EPA 608.3
Endrin	EPA 8081B
	EPA 608.3
Endrin aldehyde	EPA 8081B
	EPA 608.3
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
	EPA 608.3
Heptachlor	EPA 8081B
	EPA 608.3
Heptachlor epoxide	EPA 8081B
	EPA 608.3
Isodrin	EPA 8270E
Kepone	EPA 8081B
Lindane	EPA 8081B
	EPA 608.3
Methoxychlor	EPA 8081B
	EPA 608.3

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Chlorinated Hydrocarbon Pesticides

Mirex	EPA 8081B
	EPA 608.3
PCNB	EPA 8270E
Toxaphene	EPA 8081B
	EPA 608.3

Chlorinated Hydrocarbons

1,2,3-Trichlorobenzene	EPA 8260D
1,2,4,5-Tetrachlorobenzene	EPA 8270E
1,2,4-Trichlorobenzene	EPA 625.1
	EPA 8270E
1-Chloronaphthalene	EPA 8270E
2-Chloronaphthalene	EPA 625.1
	EPA 8270E
Hexachlorobenzene	EPA 625.1
	EPA 8270E
Hexachlorobutadiene	EPA 625.1
	EPA 8270E
Hexachlorocyclopentadiene	EPA 625.1
	EPA 8270E
Hexachloroethane	EPA 625.1
	EPA 8270E
Hexachloropropene	EPA 8270E
Pentachlorobenzene	EPA 8270E

Chlorophenoxy Acid Pesticides

2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A

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Chlorophenoxy Acid Pesticides

2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
	EPA 8270E
Pentachlorophenol	EPA 8151A

Demand

Biochemical Oxygen Demand	SM 5210B-2016
Carbonaceous BOD	SM 5210B-2016
Chemical Oxygen Demand	EPA 410.4, Rev. 2.0 (1993)

Dioxins and Furans

1,2,3,4,6,7,8,9-Octachlorodibenzofura	EPA 8290A
	EPA 1613B
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-d	EPA 8290A
	EPA 1613B
1,2,3,4,6,7,8-Heptachlorodibenzofurar	EPA 8290A
	EPA 1613B
1,2,3,4,6,7,8-Heptachlorodibenzo-p-di	EPA 8290A
	EPA 1613B
1,2,3,4,7,8,9-Heptachlorodibenzofurar	EPA 8290A
	EPA 1613B
1,2,3,4,7,8-Hexachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxi	EPA 8290A

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Dioxins and Furans

1,2,3,4,7,8-Hexachlorodibenzo-p-dioxi	EPA 1613B
1,2,3,6,7,8-Hexachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxi	EPA 8290A
	EPA 1613B
1,2,3,7,8,9-Hexachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxi	EPA 8290A
	EPA 1613B
1,2,3,7,8-Pentachlorodibenzofuran	EPA 8290A
	EPA 1613B
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	EPA 8290A
	EPA 1613B
2,3,4,6,7,8-Hexachlorodibenzofuran	EPA 8290A
	EPA 1613B
2,3,4,7,8-Pentachlorodibenzofuran	EPA 8290A
	EPA 1613B
2,3,7,8-Tetrachlorodibenzofuran	EPA 8290A
	EPA 1613B
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 8290A
	EPA 1613B

Dissolved Gases

Acetylene	RSK-175
Ethane	RSK-175
Ethene (Ethylene)	RSK-175
Methane	RSK-175
Propane	RSK-175

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Fuel Oxygenates

Di-isopropyl ether	EPA 8260D
Ethanol	EPA 1671
	EPA 8260D
	EPA 8015D
	EPA 8015C
Methyl tert-butyl ether	EPA 8260D
	EPA 624.1
tert-amyl methyl ether (TAME)	EPA 8260D
tert-butyl alcohol	EPA 1666A
	EPA 8260D
	EPA 624.1
tert-butyl ethyl ether (ETBE)	EPA 8260D

Haloethers

2,2'-Oxybis(1-chloropropane)	EPA 625.1
	EPA 8270E
4-Bromophenylphenyl ether	EPA 625.1
	EPA 8270E
4-Chlorophenylphenyl ether	EPA 625.1
	EPA 8270E
Bis(2-chloroethoxy)methane	EPA 625.1
	EPA 8270E
Bis(2-chloroethyl)ether	EPA 625.1
	EPA 8270E

Low Level Halocarbons

1,2-Dibromo-3-chloropropane, Low Le	EPA 8011
1,2-Dibromoethane, Low Level	EPA 8011

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Low Level Polynuclear Aromatics

Acenaphthene Low Level	EPA 8270E SIM
Acenaphthylene Low Level	EPA 8270E SIM
Anthracene Low Level	EPA 8270E SIM
Benzo(a)anthracene Low Level	EPA 8270E SIM
Benzo(a)pyrene Low Level	EPA 8270E SIM
Benzo(b)fluoranthene Low Level	EPA 8270E SIM
Benzo(g,h,i)perylene Low Level	EPA 8270E SIM
Benzo(k)fluoranthene Low Level	EPA 8270E SIM
Chrysene Low Level	EPA 8270E SIM
Dibenzo(a,h)anthracene Low Level	EPA 8270E SIM
Fluoranthene Low Level	EPA 8270E SIM
Fluorene Low Level	EPA 8270E SIM
Indeno(1,2,3-cd)pyrene Low Level	EPA 8270E SIM
Naphthalene Low Level	EPA 8270E SIM
Phenanthrene Low Level	EPA 8270E SIM
Pyrene Low Level	EPA 8270E SIM

Metals I

Barium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Cadmium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Calcium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D

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Metals I

Calcium, Total	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Chromium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Copper, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Iron, Total	SM 3500-Fe B-2011
	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Lead, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Magnesium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Manganese, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)

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Metals I

Nickel, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Potassium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Silver, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Sodium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Strontium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)

Metals II

Aluminum, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Antimony, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D

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Metals II

Antimony, Total	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Arsenic, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Beryllium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Chromium VI	EPA 218.6, Rev. 3.3 (1994)
	EPA 7196A
	EPA 7199
	SM 3500-Cr B-2011
Mercury, Total	EPA 245.1, Rev. 3.0 (1994)
	EPA 7470A
Selenium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Vanadium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Zinc, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B

Serial No.: 68523

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
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CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL *NY Lab Id No: 10670*
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Metals II	
Zinc, Total	EPA 200.8, Rev. 5.4 (1994)
Metals III	
Cobalt, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Molybdenum, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Thallium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Tin, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Titanium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Uranium (Mass)	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Mineral	
Acidity	SM 2310B-2011

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Mineral

Alkalinity	SM 2320B-2011
Chloride	EPA 300.0, Rev. 2.1 (1993)
	SM 4500-Cl- C-2011
	EPA 9056A
Fluoride, Total	EPA 300.0, Rev. 2.1 (1993)
	SM 4500-F- C-2011
	EPA 9056A
Hardness, Total	SM 2340C-2011
	SM 2340B-2011
Sulfate (as SO4)	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A

Miscellaneous

Boron, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
Bromide	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Color	SM 2120B-2011
Cyanide, Available	OIA-1677-09
Cyanide, Free	OIA-1677-09
Cyanide, Total	EPA 335.4, Rev. 1.0 (1993)
	EPA 9012B
	ASTM D7511-12
Formaldehyde	EPA 8315A
non-Polar Extractable Material (TPH)	EPA 1664A
	EPA 1664B
Oil and Grease Total Recoverable	EPA 1664A
	EPA 1664B

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Miscellaneous

Organic Carbon, Total	SM 5310C-2014
	EPA 9060A
Perchlorate	EPA 6850
Phenols	EPA 420.4, Rev. 1.0 (1993)
	EPA 9066
Silica, Dissolved	SM 4500-SiO2 C-2011
Specific Conductance	SM 2510B-2011
	EPA 9050A
Sulfide (as S)	SM 4500-S2- F-2011
	SM 4500-S2- D-2011
Surfactant (MBAS)	SM 5540C-2011
Turbidity	EPA 180.1, Rev. 2.0 (1993)

Nitroaromatics and Isophorone

1,3,5-Trinitrobenzene	EPA 8330B
1,3-Dinitrobenzene	EPA 8270E
	EPA 8330B
1,4-Naphthoquinone	EPA 8270E
2,4,6-Trinitrotoluene	EPA 8330B
2,4-Dinitrotoluene	EPA 625.1
	EPA 8270E
	EPA 8330B
2,6-Dinitrotoluene	EPA 625.1
	EPA 8270E
	EPA 8330B
2-Amino-4,6-dinitrotoluene	EPA 8330B
2-Nitrotoluene	EPA 8330B
3,5-Dinitroaniline	EPA 8330B

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Nitroaromatics and Isophorone

3-Nitrotoluene	EPA 8330B
4-Amino-2,6-dinitrotoluene	EPA 8330B
4-Nitroquinoline-1-oxide	EPA 8270E
4-Nitrotoluene	EPA 8330B
Hexahydro-1,3,5-trinitro-1,3,5-triazine	EPA 8330B
Isophorone	EPA 625.1
	EPA 8270E
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330B
Nitrobenzene	EPA 625.1
	EPA 8270E
	EPA 8330B
Nitroglycerine	EPA 8330B
Octahydro-tetranitro-tetrazocine	EPA 8330B
Pentaerythritol tetranitrate	EPA 8330B

Nitrosoamines

N-Nitrosodiethylamine	EPA 8270E
N-Nitrosodimethylamine	EPA 625.1
	EPA 8270E
N-Nitrosodi-n-butylamine	EPA 8270E
N-Nitrosodi-n-propylamine	EPA 625.1
	EPA 8270E
N-Nitrosodiphenylamine	EPA 625.1
	EPA 8270E
N-nitrosomethylethylamine	EPA 8270E
N-nitrosomorpholine	EPA 8270E
N-nitrosopiperidine	EPA 8270E
N-Nitrosopyrrolidine	EPA 8270E

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Nutrient

Ammonia (as N)	SM 4500-NH3 C-2011
	EPA 350.1, Rev. 2.0 (1993)
	SM 4500-NH3 D-2011 or E-2011
Kjeldahl Nitrogen, Total	EPA 351.2, Rev. 2.0 (1993)
Nitrate (as N)	EPA 353.2, Rev. 2.0 (1993)
	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Nitrate-Nitrite (as N)	EPA 353.2, Rev. 2.0 (1993)
Nitrite (as N)	EPA 353.2, Rev. 2.0 (1993)
	EPA 300.0, Rev. 2.1 (1993)
	EPA 9056A
Orthophosphate (as P)	EPA 365.3 (Issued 1978)
	SM 4500-P E-2011
Phosphorus, Total	EPA 365.1, Rev. 2.0 (1993)
	SM 4500-P (F-H)-2011

Organophosphate Pesticides

Atrazine	EPA 8270E
Dimethoate	EPA 8270E
Parathion ethyl	EPA 8270E
Parathion methyl	EPA 8270E
Phorate	EPA 8270E
Sulfotepp	EPA 8270E
Thionazin	EPA 8270E

Petroleum Hydrocarbons

Diesel Range Organics	EPA 8015D
	EPA 8015C

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Petroleum Hydrocarbons

Gasoline Range Organics	EPA 8015D
	EPA 8015C

Phthalate Esters

Benzyl butyl phthalate	EPA 625.1
	EPA 8270E
Bis(2-ethylhexyl) phthalate	EPA 625.1
	EPA 8270E
Diethyl phthalate	EPA 625.1
	EPA 8270E
Dimethyl phthalate	EPA 625.1
	EPA 8270E
Di-n-butyl phthalate	EPA 625.1
	EPA 8270E
Di-n-octyl phthalate	EPA 625.1
	EPA 8270E

Polychlorinated Biphenyls

Aroclor 1016 (PCB-1016)	EPA 8082A
	EPA 608.3
Aroclor 1221 (PCB-1221)	EPA 8082A
	EPA 608.3
Aroclor 1232 (PCB-1232)	EPA 8082A
	EPA 608.3
Aroclor 1242 (PCB-1242)	EPA 8082A
	EPA 608.3
Aroclor 1248 (PCB-1248)	EPA 8082A
	EPA 608.3

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Polychlorinated Biphenyls

Aroclor 1254 (PCB-1254)	EPA 8082A
	EPA 608.3
Aroclor 1260 (PCB-1260)	EPA 8082A
	EPA 608.3
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A
PCB 1	EPA 1668A
	EPA 1668C
PCB 10	EPA 1668A
	EPA 1668C
PCB 100	EPA 1668A
	EPA 1668C
PCB 101	EPA 1668A
	EPA 1668C
PCB 102	EPA 1668A
	EPA 1668C
PCB 103	EPA 1668A
	EPA 1668C
PCB 104	EPA 1668A
	EPA 1668C
PCB 105	EPA 1668A
	EPA 1668C
PCB 106	EPA 1668A
	EPA 1668C
PCB 107	EPA 1668A
	EPA 1668C
PCB 108	EPA 1668A

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Polychlorinated Biphenyls

PCB 108	EPA 1668C
PCB 109	EPA 1668A
	EPA 1668C
PCB 11	EPA 1668A
	EPA 1668C
PCB 110	EPA 1668A
	EPA 1668C
PCB 111	EPA 1668A
	EPA 1668C
PCB 112	EPA 1668A
	EPA 1668C
PCB 113	EPA 1668A
	EPA 1668C
PCB 114	EPA 1668A
	EPA 1668C
PCB 115	EPA 1668A
	EPA 1668C
PCB 116	EPA 1668A
	EPA 1668C
PCB 117	EPA 1668A
	EPA 1668C
PCB 118	EPA 1668A
	EPA 1668C
PCB 119	EPA 1668A
	EPA 1668C
PCB 12	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 120	EPA 1668A
	EPA 1668C
PCB 121	EPA 1668A
	EPA 1668C
PCB 122	EPA 1668A
	EPA 1668C
PCB 123	EPA 1668A
	EPA 1668C
PCB 124	EPA 1668A
	EPA 1668C
PCB 125	EPA 1668A
	EPA 1668C
PCB 126	EPA 1668A
	EPA 1668C
PCB 127	EPA 1668A
	EPA 1668C
PCB 128	EPA 1668A
	EPA 1668C
PCB 129	EPA 1668A
	EPA 1668C
PCB 13	EPA 1668A
	EPA 1668C
PCB 130	EPA 1668A
	EPA 1668C
PCB 131	EPA 1668A
	EPA 1668C
PCB 132	EPA 1668A

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Polychlorinated Biphenyls

PCB 132	EPA 1668C
PCB 133	EPA 1668A
	EPA 1668C
PCB 134	EPA 1668A
	EPA 1668C
PCB 135	EPA 1668A
	EPA 1668C
PCB 136	EPA 1668A
	EPA 1668C
PCB 137	EPA 1668A
	EPA 1668C
PCB 138	EPA 1668A
	EPA 1668C
PCB 139	EPA 1668A
	EPA 1668C
PCB 14	EPA 1668A
	EPA 1668C
PCB 140	EPA 1668A
	EPA 1668C
PCB 141	EPA 1668A
	EPA 1668C
PCB 142	EPA 1668A
	EPA 1668C
PCB 143	EPA 1668A
	EPA 1668C
PCB 144	EPA 1668A
	EPA 1668C

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PCB 145	EPA 1668A
	EPA 1668C
PCB 146	EPA 1668A
	EPA 1668C
PCB 147	EPA 1668A
	EPA 1668C
PCB 148	EPA 1668A
	EPA 1668C
PCB 149	EPA 1668A
	EPA 1668C
PCB 15	EPA 1668A
	EPA 1668C
PCB 150	EPA 1668A
	EPA 1668C
PCB 151	EPA 1668A
	EPA 1668C
PCB 152	EPA 1668A
	EPA 1668C
PCB 153	EPA 1668A
	EPA 1668C
PCB 154	EPA 1668A
	EPA 1668C
PCB 155	EPA 1668A
	EPA 1668C
PCB 156	EPA 1668A
	EPA 1668C
PCB 157	EPA 1668A

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PCB 157	EPA 1668C
PCB 158	EPA 1668A
	EPA 1668C
PCB 159	EPA 1668A
	EPA 1668C
PCB 16	EPA 1668A
	EPA 1668C
PCB 160	EPA 1668A
	EPA 1668C
PCB 161	EPA 1668A
	EPA 1668C
PCB 162	EPA 1668A
	EPA 1668C
PCB 163	EPA 1668A
	EPA 1668C
PCB 164	EPA 1668A
	EPA 1668C
PCB 165	EPA 1668A
	EPA 1668C
PCB 166	EPA 1668A
	EPA 1668C
PCB 167	EPA 1668A
	EPA 1668C
PCB 168	EPA 1668A
	EPA 1668C
PCB 169	EPA 1668A
	EPA 1668C

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PCB 17	EPA 1668A
	EPA 1668C
PCB 170	EPA 1668A
	EPA 1668C
PCB 171	EPA 1668A
	EPA 1668C
PCB 172	EPA 1668A
	EPA 1668C
PCB 173	EPA 1668A
	EPA 1668C
PCB 174	EPA 1668A
	EPA 1668C
PCB 175	EPA 1668A
	EPA 1668C
PCB 176	EPA 1668A
	EPA 1668C
PCB 177	EPA 1668A
	EPA 1668C
PCB 178	EPA 1668A
	EPA 1668C
PCB 179	EPA 1668A
	EPA 1668C
PCB 18	EPA 1668A
	EPA 1668C
PCB 180	EPA 1668A
	EPA 1668C
PCB 181	EPA 1668A

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL *NY Lab Id No: 10670*
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Polychlorinated Biphenyls

PCB 181	EPA 1668C
PCB 182	EPA 1668A
	EPA 1668C
PCB 183	EPA 1668A
	EPA 1668C
PCB 184	EPA 1668A
	EPA 1668C
PCB 185	EPA 1668A
	EPA 1668C
PCB 186	EPA 1668A
	EPA 1668C
PCB 187	EPA 1668A
	EPA 1668C
PCB 188	EPA 1668A
	EPA 1668C
PCB 189	EPA 1668A
	EPA 1668C
PCB 19	EPA 1668A
	EPA 1668C
PCB 190	EPA 1668A
	EPA 1668C
PCB 191	EPA 1668A
	EPA 1668C
PCB 192	EPA 1668A
	EPA 1668C
PCB 193	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 194	EPA 1668A
	EPA 1668C
PCB 195	EPA 1668A
	EPA 1668C
PCB 196	EPA 1668A
	EPA 1668C
PCB 197	EPA 1668A
	EPA 1668C
PCB 198	EPA 1668A
	EPA 1668C
PCB 199	EPA 1668A
	EPA 1668C
PCB 2	EPA 1668A
	EPA 1668C
PCB 20	EPA 1668A
	EPA 1668C
PCB 200	EPA 1668A
	EPA 1668C
PCB 201	EPA 1668A
	EPA 1668C
PCB 202	EPA 1668A
	EPA 1668C
PCB 203	EPA 1668A
	EPA 1668C
PCB 204	EPA 1668A
	EPA 1668C
PCB 205	EPA 1668A

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Polychlorinated Biphenyls

PCB 205	EPA 1668C
PCB 206	EPA 1668A
	EPA 1668C
PCB 207	EPA 1668A
	EPA 1668C
PCB 208	EPA 1668A
	EPA 1668C
PCB 209	EPA 1668A
	EPA 1668C
PCB 21	EPA 1668A
	EPA 1668C
PCB 22	EPA 1668A
	EPA 1668C
PCB 23	EPA 1668A
	EPA 1668C
PCB 24	EPA 1668A
	EPA 1668C
PCB 25	EPA 1668A
	EPA 1668C
PCB 26	EPA 1668A
	EPA 1668C
PCB 27	EPA 1668A
	EPA 1668C
PCB 28	EPA 1668A
	EPA 1668C
PCB 29	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 3	EPA 1668A
	EPA 1668C
PCB 30	EPA 1668A
	EPA 1668C
PCB 31	EPA 1668A
	EPA 1668C
PCB 32	EPA 1668A
	EPA 1668C
PCB 33	EPA 1668A
	EPA 1668C
PCB 34	EPA 1668A
	EPA 1668C
PCB 35	EPA 1668A
	EPA 1668C
PCB 36	EPA 1668A
	EPA 1668C
PCB 37	EPA 1668A
	EPA 1668C
PCB 38	EPA 1668A
	EPA 1668C
PCB 39	EPA 1668A
	EPA 1668C
PCB 4	EPA 1668A
	EPA 1668C
PCB 40	EPA 1668A
	EPA 1668C
PCB 41	EPA 1668A

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Polychlorinated Biphenyls

PCB 41	EPA 1668C
PCB 42	EPA 1668A
	EPA 1668C
PCB 43	EPA 1668A
	EPA 1668C
PCB 44	EPA 1668A
	EPA 1668C
PCB 45	EPA 1668A
	EPA 1668C
PCB 46	EPA 1668A
	EPA 1668C
PCB 47	EPA 1668A
	EPA 1668C
PCB 48	EPA 1668A
	EPA 1668C
PCB 49	EPA 1668A
	EPA 1668C
PCB 5	EPA 1668A
	EPA 1668C
PCB 50	EPA 1668A
	EPA 1668C
PCB 51	EPA 1668A
	EPA 1668C
PCB 52	EPA 1668A
	EPA 1668C
PCB 53	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 54	EPA 1668A
	EPA 1668C
PCB 55	EPA 1668A
	EPA 1668C
PCB 56	EPA 1668A
	EPA 1668C
PCB 57	EPA 1668A
	EPA 1668C
PCB 58	EPA 1668A
	EPA 1668C
PCB 59	EPA 1668A
	EPA 1668C
PCB 6	EPA 1668A
	EPA 1668C
PCB 60	EPA 1668A
	EPA 1668C
PCB 61	EPA 1668A
	EPA 1668C
PCB 62	EPA 1668A
	EPA 1668C
PCB 63	EPA 1668A
	EPA 1668C
PCB 64	EPA 1668A
	EPA 1668C
PCB 65	EPA 1668A
	EPA 1668C
PCB 66	EPA 1668A

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Polychlorinated Biphenyls

PCB 66	EPA 1668C
PCB 67	EPA 1668A
	EPA 1668C
PCB 68	EPA 1668A
	EPA 1668C
PCB 69	EPA 1668A
	EPA 1668C
PCB 7	EPA 1668A
	EPA 1668C
PCB 70	EPA 1668A
	EPA 1668C
PCB 71	EPA 1668A
	EPA 1668C
PCB 72	EPA 1668A
	EPA 1668C
PCB 73	EPA 1668A
	EPA 1668C
PCB 74	EPA 1668A
	EPA 1668C
PCB 75	EPA 1668A
	EPA 1668C
PCB 76	EPA 1668A
	EPA 1668C
PCB 77	EPA 1668A
	EPA 1668C
PCB 78	EPA 1668A
	EPA 1668C

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PCB 79	EPA 1668A
	EPA 1668C
PCB 8	EPA 1668A
	EPA 1668C
PCB 80	EPA 1668A
	EPA 1668C
PCB 81	EPA 1668A
	EPA 1668C
PCB 82	EPA 1668A
	EPA 1668C
PCB 83	EPA 1668A
	EPA 1668C
PCB 84	EPA 1668A
	EPA 1668C
PCB 85	EPA 1668A
	EPA 1668C
PCB 86	EPA 1668A
	EPA 1668C
PCB 87	EPA 1668A
	EPA 1668C
PCB 88	EPA 1668A
	EPA 1668C
PCB 89	EPA 1668A
	EPA 1668C
PCB 9	EPA 1668A
	EPA 1668C
PCB 90	EPA 1668A

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PCB 90	EPA 1668C
PCB 91	EPA 1668A
	EPA 1668C
PCB 92	EPA 1668A
	EPA 1668C
PCB 93	EPA 1668A
	EPA 1668C
PCB 94	EPA 1668A
	EPA 1668C
PCB 95	EPA 1668A
	EPA 1668C
PCB 96	EPA 1668A
	EPA 1668C
PCB 97	EPA 1668A
	EPA 1668C
PCB 98	EPA 1668A
	EPA 1668C
PCB 99	EPA 1668A
	EPA 1668C
PCB Congeners, Total	EPA 1668C

Polynuclear Aromatics

2-Acetylaminofluorene	EPA 8270E
3-Methylcholanthrene	EPA 8270E
7,12-Dimethylbenzyl (a) anthracene	EPA 8270E
Acenaphthene	EPA 625.1
	EPA 8270E
Acenaphthylene	EPA 625.1

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Polynuclear Aromatics

Acenaphthylene	EPA 8270E
Anthracene	EPA 625.1
	EPA 8270E
Benzo(a)anthracene	EPA 625.1
	EPA 8270E
Benzo(a)pyrene	EPA 625.1
	EPA 8270E
Benzo(b)fluoranthene	EPA 625.1
	EPA 8270E
Benzo(g,h,i)perylene	EPA 625.1
	EPA 8270E
Benzo(k)fluoranthene	EPA 625.1
	EPA 8270E
Chrysene	EPA 625.1
	EPA 8270E
Dibenzo(a,h)anthracene	EPA 625.1
	EPA 8270E
Fluoranthene	EPA 625.1
	EPA 8270E
Fluorene	EPA 625.1
	EPA 8270E
Indeno(1,2,3-cd)pyrene	EPA 625.1
	EPA 8270E
Naphthalene	EPA 625.1
	EPA 8270E
Phenanthrene	EPA 625.1
	EPA 8270E

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Polynuclear Aromatics

Pyrene	EPA 625.1
	EPA 8270E

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270E
2,4,5-Trichlorophenol	EPA 625.1
	EPA 8270E
2,4,6-Trichlorophenol	EPA 625.1
	EPA 8270E
2,4-Dichlorophenol	EPA 625.1
	EPA 8270E
2,4-Dimethylphenol	EPA 625.1
	EPA 8270E
2,4-Dinitrophenol	EPA 625.1
	EPA 8270E
2,6-Dichlorophenol	EPA 8270E
2-Chlorophenol	EPA 625.1
	EPA 8270E
2-Methyl-4,6-dinitrophenol	EPA 625.1
	EPA 8270E
2-Methylphenol	EPA 625.1
	EPA 8270E
2-Nitrophenol	EPA 625.1
	EPA 8270E
3-Methylphenol	EPA 625.1
	EPA 8270E
4-Chloro-3-methylphenol	EPA 625.1
	EPA 8270E

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Priority Pollutant Phenols

4-Methylphenol	EPA 625.1
	EPA 8270E
4-Nitrophenol	EPA 625.1
	EPA 8270E
Pentachlorophenol	EPA 625.1
	EPA 8270E
Phenol	EPA 625.1
	EPA 8270E

Residue

Settleable Solids	SM 2540 F-2015
Solids, Total	SM 2540 B-2015
Solids, Total Dissolved	SM 2540 C-2015
Solids, Total Suspended	SM 2540 D-2015

Semi-Volatile Organics

1,1'-Biphenyl	EPA 8270E
1,2-Dichlorobenzene, Semi-volatile	EPA 8270E
1,3-Dichlorobenzene, Semi-volatile	EPA 8270E
1,4-Dichlorobenzene, Semi-volatile	EPA 8270E
2-Methylnaphthalene	EPA 625.1
	EPA 8270E
2-Picoline	EPA 8270E
4-Amino biphenyl	EPA 8270E
Acetophenone	EPA 625.1
	EPA 8270E
alpha-Terpineol	EPA 625.1
Benzaldehyde	EPA 8270E

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Semi-Volatile Organics

Benzoic Acid	EPA 8270E
Benzyl alcohol	EPA 8270E
Caprolactam	EPA 8270E
Dibenzofuran	EPA 8270E
Ethyl methanesulfonate	EPA 8270E
Isosafrole	EPA 8270E
Methyl methanesulfonate	EPA 8270E
n-Decane	EPA 625.1
n-Octadecane	EPA 625.1
O,O,O-Triethyl phosphorothioate	EPA 8270E
p-Dimethylaminoazobenzene	EPA 8270E
Phenacetin	EPA 8270E
Safrole	EPA 8270E

Volatile Aromatics

1,2,4-Trichlorobenzene, Volatile	EPA 8260D
1,2,4-Trimethylbenzene	EPA 8260D
1,2-Dichlorobenzene	EPA 8260D
	EPA 624.1
	EPA 524.2
1,3,5-Trimethylbenzene	EPA 8260D
1,3-Dichlorobenzene	EPA 8260D
	EPA 624.1
1,4-Dichlorobenzene	EPA 8260D
	EPA 624.1
2-Chlorotoluene	EPA 8260D
4-Chlorotoluene	EPA 8260D
Benzene	EPA 8260D

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WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL
EUROFINS LANCASTER LABORATORIES ENVIRONMENT TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

NY Lab Id No: 10670

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Volatile Aromatics

Benzene	EPA 624.1
	EPA 524.2
Bromobenzene	EPA 8260D
Chlorobenzene	EPA 8260D
	EPA 624.1
	EPA 524.2
Ethyl benzene	EPA 8260D
	EPA 624.1
Isopropylbenzene	EPA 8260D
m/p-Xylenes	EPA 8260D
	EPA 624.1
Naphthalene, Volatile	EPA 8260D
	EPA 624.1
n-Butylbenzene	EPA 8260D
n-Propylbenzene	EPA 8260D
o-Xylene	EPA 8260D
	EPA 624.1
p-Isopropyltoluene (P-Cymene)	EPA 8260D
sec-Butylbenzene	EPA 8260D
Styrene	EPA 8260D
	EPA 624.1
tert-Butylbenzene	EPA 8260D
Toluene	EPA 8260D
	EPA 624.1
	EPA 524.2
Total Xylenes	EPA 1666A
	EPA 8260D

Serial No.: 68523

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All approved analytes are listed below:

Volatile Aromatics	
Total Xylenes	EPA 624.1
Volatile Chlorinated Organics	
Benzyl chloride	EPA 8260D
Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 8260D
1,1,1-Trichloroethane	EPA 8260D
	EPA 624.1
1,1,2,2-Tetrachloroethane	EPA 8260D
	EPA 624.1
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260D
	EPA 624.1
1,1,2-Trichloroethane	EPA 8260D
	EPA 624.1
1,1-Dichloroethane	EPA 8260D
	EPA 624.1
1,1-Dichloroethene	EPA 8260D
	EPA 624.1
1,1-Dichloropropene	EPA 8260D
1,2,3-Trichloropropane	EPA 8260D
1,2-Dibromo-3-chloropropane	EPA 8260D
1,2-Dibromoethane	EPA 8260D
1,2-Dichloro-1,1,2-Trifluoroethane	EPA 8260D
1,2-Dichloroethane	EPA 8260D
	EPA 624.1
	EPA 524.2
1,2-Dichloropropane	EPA 8260D

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Volatile Halocarbons

1,2-Dichloropropane	EPA 624.1
1,3-Dichloropropane	EPA 8260D
2,2-Dichloropropane	EPA 8260D
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260D
2-Chloroethylvinyl ether	EPA 8260D
	EPA 624.1
3-Chloropropene (Allyl chloride)	EPA 8260D
Bromochloromethane	EPA 8260D
Bromodichloromethane	EPA 8260D
	EPA 624.1
Bromoform	EPA 8260D
	EPA 624.1
Bromomethane	EPA 8260D
	EPA 624.1
Carbon tetrachloride	EPA 8260D
	EPA 624.1
Chloroethane	EPA 8260D
	EPA 624.1
Chloroform	EPA 8260D
	EPA 624.1
	EPA 524.2
Chloromethane	EPA 8260D
	EPA 624.1
cis-1,2-Dichloroethene	EPA 8260D
	EPA 624.1
cis-1,3-Dichloropropene	EPA 8260D
	EPA 624.1

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Volatile Halocarbons

Dibromochloromethane	EPA 8260D
	EPA 624.1
Dibromomethane	EPA 8260D
Dichlorodifluoromethane	EPA 8260D
	EPA 624.1
Hexachlorobutadiene, Volatile	EPA 8260D
Methyl iodide	EPA 8260D
Methylene chloride	EPA 8260D
	EPA 624.1
	EPA 524.2
Tetrachloroethene	EPA 8260D
	EPA 624.1
trans-1,2-Dichloroethene	EPA 8260D
	EPA 624.1
trans-1,3-Dichloropropene	EPA 8260D
	EPA 624.1
trans-1,4-Dichloro-2-butene	EPA 8260D
Trichloroethene	EPA 8260D
	EPA 624.1
Trichlorofluoromethane	EPA 8260D
	EPA 624.1
Vinyl chloride	EPA 8260D
	EPA 624.1

Volatiles Organics

1,4-Dioxane	EPA 8260D
	EPA 8260D SIM
	EPA 8270E

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Volatiles Organics

1,4-Dioxane	EPA 8270E SIM
2-Butanone (Methylethyl ketone)	EPA 8260D
2-Hexanone	EPA 8260D
2-Nitropropane	EPA 8260D
4-Methyl-2-Pentanone	EPA 1666A
	EPA 8260D
	EPA 624.1
	EPA 524.2
Acetone	EPA 8260D
	EPA 624.1
	EPA 524.2
Acetonitrile	EPA 1671
	EPA 8260D
	EPA 624.1
Carbon Disulfide	EPA 8260D
Cyclohexane	EPA 8260D
Di-ethyl ether	EPA 8260D
Diethylamine	EPA 1671
Dimethyl sulfoxide	EPA 1671
Ethyl Acetate	EPA 1666A
	EPA 8260D
	EPA 624.1
Ethylene Glycol	EPA 8015D
	EPA 8015C
Hexane	EPA 1666A
Isobutyl alcohol	EPA 8260D
	EPA 8015D

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Volatiles Organics

Isobutyl alcohol	EPA 8015C
Isobutyraldehyde	EPA 1666A
Isopropanol	EPA 1666A
	EPA 8260D
Isopropyl Acetate	EPA 1666A
Methanol	EPA 1671
	EPA 8015D
	EPA 8015C
Methyl acetate	EPA 8260D
Methyl cellosolve (2-Methoxyethanol)	EPA 1671
Methyl cyclohexane	EPA 8260D
Methyl formate	EPA 1666A
n-Amyl Acetate	EPA 1666A
n-Amyl alcohol	EPA 1666A
n-Butanol	EPA 8260D
n-Butyl Acetate	EPA 1666A
n-Heptane	EPA 1666A
n-Propanol	EPA 1671
o-Toluidine	EPA 8270E
Propylene Glycol	EPA 8015D
Tetrahydrofuran	EPA 1666A
	EPA 8260D
	EPA 624.1
	EPA 524.2
Triethylamine	EPA 1671
Vinyl acetate	EPA 8260D
	EPA 624.1

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Sample Preparation Methods

SM 4500-P B(5)-2011
EPA 5030C
EPA 3010A
EPA 3005A
EPA 3510C
EPA 3520C
SM 4500-NH3 B-2011
SM 4500-F B-2011

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is hereby APPROVED as an Environmental Laboratory for the category
ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved subcategories and/or analytes are listed below:

Chlorinated Hydrocarbon Pesticides

Kepone	EPA 8270E

Miscellaneous

Turbidity	SM 2130 B-2011

Nitroaromatics and Isophorone

1,3,5-Trinitrobenzene	EPA 8270E

Organophosphate Pesticides

Disulfoton	EPA 8270E
Famphur	EPA 8270E

Perfluorinated Alkyl Acids

11CL-PF3OUDS	EPA 1633 (Draft)
4:2FTS	EPA 1633 (Draft)
6:2FTS	EPA 1633 (Draft)
8:2FTS	EPA 1633 (Draft)
9CL-PF3ONS	EPA 1633 (Draft)
ADONA	EPA 1633 (Draft)
Hexafluoropropylene Oxide Dimer Aci	EPA 1633 (Draft)
NETFOSAA	EPA 1633 (Draft)
NMEFOSAA	EPA 1633 (Draft)
Nonafluoro-3,6-Dioxaheptanoic Acid	EPA 1633 (Draft)
Perflourotridecanoic Acid (PFTRDA)	EPA 1633 (Draft)
Perfluordecanoic Acid (PFDA)	EPA 1633 (Draft)
Perfluoro-3-Methoxypropanoic Acid	EPA 1633 (Draft)
Perfluoro-4-Methoxybutanoic Acid	EPA 1633 (Draft)
Perfluorobutanesulfonic Acid (PFBS)	EPA 1633 (Draft)
Perfluorobutanoic Acid (PFBA)	EPA 1633 (Draft)
Perfluorododecanoic Acid (PFDOA)	EPA 1633 (Draft)

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Perfluorinated Alkyl Acids

Perfluoroheptanesulfonic Acid (PFHPS	EPA 1633 (Draft)
Perfluoroheptanoic Acid (PFHPA)	EPA 1633 (Draft)
Perfluorohexanesulfonic Acid (PFHXS	EPA 1633 (Draft)
Perfluorohexanoic Acid (PFHXA)	EPA 1633 (Draft)
Perfluorononanoic Acid (PFNA)	EPA 1633 (Draft)
Perfluorooctanesulfonic Acid (PFOS)	EPA 1633 (Draft)
Perfluorooctanoic Acid (PFOA)	EPA 1633 (Draft)
Perfluoropentanesulfonic Acid (PFPES	EPA 1633 (Draft)
Perfluoropentanoic Acid (PFPEA)	EPA 1633 (Draft)
Perfluorotetradecanoic Acid (PFTA)	EPA 1633 (Draft)
Perfluoroundecanoic Acid (PFUNA)	EPA 1633 (Draft)
PFEESA	EPA 1633 (Draft)

Semi-Volatile Organics

alpha-Terpineol	EPA 8270E
Aramite	EPA 8270E

Sample Preparation Methods

	EPA 3511

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All approved analytes are listed below:

Acrylates

Acrolein (Propenal)	EPA 8260D
Acrylonitrile	EPA 8260D
Ethyl methacrylate	EPA 8260D
Methyl acrylonitrile	EPA 8260D
Methyl methacrylate	EPA 8260D

Amines

1,2-Diphenylhydrazine	EPA 8270E
1-Naphthylamine	EPA 8270E
2-Naphthylamine	EPA 8270E
2-Nitroaniline	EPA 8270E
3-Nitroaniline	EPA 8270E
4,4'-Methylenebis(2-chloroaniline)	EPA 8270E
4-Chloroaniline	EPA 8270E
4-Nitroaniline	EPA 8270E
5-Nitro-o-toluidine	EPA 8270E
Aniline	EPA 8270E
Carbazole	EPA 8270E
Diphenylamine	EPA 8270E
Methapyrilene	EPA 8270E
Pronamide	EPA 8270E

Benzidines

3,3'-Dichlorobenzidine	EPA 8270E
3,3'-Dimethylbenzidine	EPA 8270E
Benzidine	EPA 8270E

Carbamate Pesticides

Aldicarb	EPA 8318A

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Carbamate Pesticides

Aldicarb Sulfone	EPA 8318A
Carbofuran	EPA 8318A

Characteristic Testing

Corrosivity (pH)	EPA 9045D
Free Liquids	EPA 9095B
Ignitability	EPA 1010B
	EPA 1010A
Synthetic Precipitation Leaching Proc.	EPA 1312
TCLP	EPA 1311

Chlorinated Hydrocarbon Pesticides

2,4'-DDD (Mitotane)	EPA 8081B
4,4'-DDD	EPA 8081B
4,4'-DDE	EPA 8081B
4,4'-DDT	EPA 8081B
Aldrin	EPA 8081B
alpha-BHC	EPA 8081B
alpha-Chlordane	EPA 8081B
Atrazine	EPA 8270E
beta-BHC	EPA 8081B
Chlordane Total	EPA 8081B
Chlorobenzilate	EPA 8270E
delta-BHC	EPA 8081B
Diallate	EPA 8270E
Dieldrin	EPA 8081B
Endosulfan I	EPA 8081B
Endosulfan II	EPA 8081B

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Chlorinated Hydrocarbon Pesticides

Endosulfan sulfate	EPA 8081B
Endrin	EPA 8081B
Endrin aldehyde	EPA 8081B
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B
Heptachlor epoxide	EPA 8081B
Isodrin	EPA 8270E
Kepone	EPA 8081B
Lindane	EPA 8081B
Methoxychlor	EPA 8081B
Mirex	EPA 8081B
Pentachloronitrobenzene	EPA 8270E
Toxaphene	EPA 8081B

Chlorinated Hydrocarbons

1,2,3-Trichlorobenzene	EPA 8260D
1,2,4,5-Tetrachlorobenzene	EPA 8270E
1,2,4-Trichlorobenzene	EPA 8270E
1-Chloronaphthalene	EPA 8270E
2-Chloronaphthalene	EPA 8270E
Hexachlorobenzene	EPA 8270E
Hexachlorobutadiene	EPA 8270E
Hexachlorocyclopentadiene	EPA 8270E
Hexachloroethane	EPA 8270E
Hexachloropropene	EPA 8270E
Pentachlorobenzene	EPA 8270E

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Chlorophenoxy Acid Pesticides

2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
MCPA	EPA 8151A
MCPP	EPA 8151A
Pentachlorophenol	EPA 8151A

Dioxins and Furans

1,2,3,4,6,7,8,9-Octachlorodibenzofura	EPA 8290A
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-d	EPA 8290A
1,2,3,4,6,7,8-Heptachlorodibenzofurar	EPA 8290A
1,2,3,4,6,7,8-Heptachlorodibenzo-p-di	EPA 8290A
1,2,3,4,7,8,9-Heptachlorodibenzofurar	EPA 8290A
1,2,3,4,7,8-Hexachlorodibenzofuran	EPA 8290A
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxi	EPA 8290A
1,2,3,6,7,8-Hexachlorodibenzofuran	EPA 8290A
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxi	EPA 8290A
1,2,3,7,8,9-Hexachlorodibenzofuran	EPA 8290A
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxi	EPA 8290A
1,2,3,7,8-Pentachlorodibenzofuran	EPA 8290A
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	EPA 8290A
2,3,4,6,7,8-Hexachlorodibenzofuran	EPA 8290A
2,3,4,7,8-Pentachlorodibenzofuran	EPA 8290A

Serial No.: 68525

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL *NY Lab Id No: 10670*
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Dioxins and Furans

2,3,7,8-Tetrachlorodibenzofuran	EPA 8290A
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 8290A

Haloethers

2,2'-Oxybis(1-chloropropane)	EPA 8270E
4-Bromophenylphenyl ether	EPA 8270E
4-Chlorophenylphenyl ether	EPA 8270E
Bis(2-chloroethoxy)methane	EPA 8270E
Bis(2-chloroethyl)ether	EPA 8270E

Low Level Polynuclear Aromatic Hydrocarbons

Acenaphthene Low Level	EPA 8270E SIM
Acenaphthylene Low Level	EPA 8270E SIM
Anthracene Low Level	EPA 8270E SIM
Benzo(a)anthracene Low Level	EPA 8270E SIM
Benzo(a)pyrene Low Level	EPA 8270E SIM
Benzo(b)fluoranthene Low Level	EPA 8270E SIM
Benzo(g,h,i)perylene Low Level	EPA 8270E SIM
Benzo(k)fluoranthene Low Level	EPA 8270E SIM
Chrysene Low Level	EPA 8270E SIM
Dibenzo(a,h)anthracene Low Level	EPA 8270E SIM
Fluoranthene Low Level	EPA 8270E SIM
Fluorene Low Level	EPA 8270E SIM
Indeno(1,2,3-cd)pyrene Low Level	EPA 8270E SIM
Naphthalene Low Level	EPA 8270E SIM
Phenanthrene Low Level	EPA 8270E SIM
Pyrene Low Level	EPA 8270E SIM

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Metals I

Barium, Total	EPA 6010D
	EPA 6020B
Cadmium, Total	EPA 6010D
	EPA 6020B
Calcium, Total	EPA 6010D
	EPA 6020B
Chromium, Total	EPA 6010D
	EPA 6020B
Copper, Total	EPA 6010D
	EPA 6020B
Iron, Total	EPA 6010D
	EPA 6020B
Lead, Total	EPA 6010D
	EPA 6020B
Magnesium, Total	EPA 6010D
	EPA 6020B
Manganese, Total	EPA 6010D
	EPA 6020B
Nickel, Total	EPA 6010D
	EPA 6020B
Potassium, Total	EPA 6010D
	EPA 6020B
Silver, Total	EPA 6010D
	EPA 6020B
Sodium, Total	EPA 6010D
	EPA 6020B
Strontium, Total	EPA 6010D

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Metals I

Strontium, Total	EPA 6020B

Metals II

Aluminum, Total	EPA 6010D
	EPA 6020B
Antimony, Total	EPA 6010D
	EPA 6020B
Arsenic, Total	EPA 6010D
	EPA 6020B
Beryllium, Total	EPA 6010D
	EPA 6020B
Chromium VI	EPA 7196A
	EPA 7199
Lithium, Total	EPA 6010D
Mercury, Total	EPA 7471B
Selenium, Total	EPA 6010D
	EPA 6020B
Vanadium, Total	EPA 6010D
	EPA 6020B
Zinc, Total	EPA 6010D
	EPA 6020B

Metals III

Cobalt, Total	EPA 6010D
	EPA 6020B
Molybdenum, Total	EPA 6010D
	EPA 6020B
Silica, Dissolved	EPA 6010D

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Metals III

Thallium, Total	EPA 6010D
	EPA 6020B
Tin, Total	EPA 6010D
	EPA 6020B
Titanium, Total	EPA 6010D
	EPA 6020B

Minerals

Chloride	EPA 9056A
Fluoride, Total	EPA 9056A
Sulfate (as SO4)	EPA 9056A

Miscellaneous

Boron, Total	EPA 6010D
Cyanide, Total	EPA 9012B
Formaldehyde	EPA 8315A
Organic Carbon, Total	Lloyd Kahn Method
Perchlorate	EPA 6850
Specific Conductance	EPA 9050A

Nitroaromatics and Isophorone

1,3,5-Trinitrobenzene	EPA 8330B
1,3-Dinitrobenzene	EPA 8270E
	EPA 8330B
1,4-Dinitrobenzene	EPA 8270E
1,4-Naphthoquinone	EPA 8270E
2,4,6-Trinitrotoluene	EPA 8330B
2,4-Dinitrotoluene	EPA 8270E
	EPA 8330B

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Nitroaromatics and Isophorone

2,6-Dinitrotoluene	EPA 8270E
	EPA 8330B
2-Amino-4,6-dinitrotoluene	EPA 8330B
2-Nitrotoluene	EPA 8330B
3,5-Dinitroaniline	EPA 8330B
3-Nitrotoluene	EPA 8330B
4-Amino-2,6-dinitrotoluene	EPA 8330B
4-Dimethylaminoazobenzene	EPA 8270E
4-Nitroquinoline-1-oxide	EPA 8270E
4-Nitrotoluene	EPA 8330B
Hexahydro-1,3,5-trinitro-1,3,5-triazine	EPA 8330B
Isophorone	EPA 8270E
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330B
Nitrobenzene	EPA 8270E
	EPA 8330B
Nitroglycerine	EPA 8330B
Octahydro-tetranitro-tetrazocine	EPA 8330B
Pentaerythritol tetranitrate	EPA 8330B
Pyridine	EPA 8270E

Nitrosoamines

N-Nitrosodiethylamine	EPA 8270E
N-Nitrosodimethylamine	EPA 8270E
N-Nitrosodi-n-butylamine	EPA 8270E
N-Nitrosodi-n-propylamine	EPA 8270E
N-Nitrosodiphenylamine	EPA 8270E
N-nitrosomethylethylamine	EPA 8270E
N-nitrosomorpholine	EPA 8270E

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Nitrosoamines	
N-nitrosopiperidine	EPA 8270E
N-Nitrosopyrrolidine	EPA 8270E
Nutrients	
Nitrate (as N)	EPA 9056A
Nitrite (as N)	EPA 9056A
Organophosphate Pesticides	
Dimethoate	EPA 8270E
Parathion ethyl	EPA 8270E
Parathion methyl	EPA 8270E
Phorate	EPA 8270E
Sulfotepp	EPA 8270E
Thionazin	EPA 8270E
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015D
	EPA 8015C
Gasoline Range Organics	EPA 8015D
	EPA 8015C
Oil and Grease Total Recoverable	EPA 9071B (Solvent:Hexane)
Phthalate Esters	
Benzyl butyl phthalate	EPA 8270E
Bis(2-ethylhexyl) phthalate	EPA 8270E
Diethyl phthalate	EPA 8270E
Dimethyl phthalate	EPA 8270E
Di-n-butyl phthalate	EPA 8270E
Di-n-octyl phthalate	EPA 8270E

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All approved analytes are listed below:

Polychlorinated Biphenyls

Aroclor 1016 (PCB-1016)	EPA 8082A
Aroclor 1016 (PCB-1016) in Oil	EPA 8082A
Aroclor 1221 (PCB-1221)	EPA 8082A
Aroclor 1221 (PCB-1221) in Oil	EPA 8082A
Aroclor 1232 (PCB-1232)	EPA 8082A
Aroclor 1232 (PCB-1232) in Oil	EPA 8082A
Aroclor 1242 (PCB-1242)	EPA 8082A
Aroclor 1242 (PCB-1242) in Oil	EPA 8082A
Aroclor 1248 (PCB-1248)	EPA 8082A
Aroclor 1248 (PCB-1248) in Oil	EPA 8082A
Aroclor 1254 (PCB-1254)	EPA 8082A
Aroclor 1254 (PCB-1254) in Oil	EPA 8082A
Aroclor 1260 (PCB-1260)	EPA 8082A
Aroclor 1260 (PCB-1260) in Oil	EPA 8082A
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A
PCB 1	EPA 1668A
	EPA 1668C
PCB 10	EPA 1668A
	EPA 1668C
PCB 100	EPA 1668A
	EPA 1668C
PCB 101	EPA 1668A
	EPA 1668C
PCB 102	EPA 1668A
	EPA 1668C
PCB 103	EPA 1668A

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Polychlorinated Biphenyls

PCB 103	EPA 1668C
PCB 104	EPA 1668A
	EPA 1668C
PCB 105	EPA 1668A
	EPA 1668C
PCB 106	EPA 1668A
	EPA 1668C
PCB 107	EPA 1668A
	EPA 1668C
PCB 108	EPA 1668A
	EPA 1668C
PCB 109	EPA 1668A
	EPA 1668C
PCB 11	EPA 1668A
	EPA 1668C
PCB 110	EPA 1668A
	EPA 1668C
PCB 111	EPA 1668A
	EPA 1668C
PCB 112	EPA 1668A
	EPA 1668C
PCB 113	EPA 1668A
	EPA 1668C
PCB 114	EPA 1668A
	EPA 1668C
PCB 115	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 116	EPA 1668A
	EPA 1668C
PCB 117	EPA 1668A
	EPA 1668C
PCB 118	EPA 1668A
	EPA 1668C
PCB 119	EPA 1668A
	EPA 1668C
PCB 12	EPA 1668A
	EPA 1668C
PCB 120	EPA 1668A
	EPA 1668C
PCB 121	EPA 1668A
	EPA 1668C
PCB 122	EPA 1668A
	EPA 1668C
PCB 123	EPA 1668A
	EPA 1668C
PCB 124	EPA 1668A
	EPA 1668C
PCB 125	EPA 1668A
	EPA 1668C
PCB 126	EPA 1668A
	EPA 1668C
PCB 127	EPA 1668A
	EPA 1668C
PCB 128	EPA 1668A

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Polychlorinated Biphenyls

PCB 128	EPA 1668C
PCB 129	EPA 1668A
	EPA 1668C
PCB 13	EPA 1668A
	EPA 1668C
PCB 130	EPA 1668A
	EPA 1668C
PCB 131	EPA 1668A
	EPA 1668C
PCB 132	EPA 1668A
	EPA 1668C
PCB 133	EPA 1668A
	EPA 1668C
PCB 134	EPA 1668A
	EPA 1668C
PCB 135	EPA 1668A
	EPA 1668C
PCB 136	EPA 1668A
	EPA 1668C
PCB 137	EPA 1668A
	EPA 1668C
PCB 138	EPA 1668A
	EPA 1668C
PCB 139	EPA 1668A
	EPA 1668C
PCB 14	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 140	EPA 1668A
	EPA 1668C
PCB 141	EPA 1668A
	EPA 1668C
PCB 142	EPA 1668A
	EPA 1668C
PCB 143	EPA 1668A
	EPA 1668C
PCB 144	EPA 1668A
	EPA 1668C
PCB 145	EPA 1668A
	EPA 1668C
PCB 146	EPA 1668A
	EPA 1668C
PCB 147	EPA 1668A
	EPA 1668C
PCB 148	EPA 1668A
	EPA 1668C
PCB 149	EPA 1668A
	EPA 1668C
PCB 15	EPA 1668A
	EPA 1668C
PCB 150	EPA 1668A
	EPA 1668C
PCB 151	EPA 1668A
	EPA 1668C
PCB 152	EPA 1668A

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Polychlorinated Biphenyls

PCB 152	EPA 1668C
PCB 153	EPA 1668A
	EPA 1668C
PCB 154	EPA 1668A
	EPA 1668C
PCB 155	EPA 1668A
	EPA 1668C
PCB 156	EPA 1668A
	EPA 1668C
PCB 157	EPA 1668A
	EPA 1668C
PCB 158	EPA 1668A
	EPA 1668C
PCB 159	EPA 1668A
	EPA 1668C
PCB 16	EPA 1668A
	EPA 1668C
PCB 160	EPA 1668A
	EPA 1668C
PCB 161	EPA 1668A
	EPA 1668C
PCB 162	EPA 1668A
	EPA 1668C
PCB 163	EPA 1668A
	EPA 1668C
PCB 164	EPA 1668A
	EPA 1668C

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL *NY Lab Id No: 10670*
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Polychlorinated Biphenyls

PCB 165	EPA 1668A
	EPA 1668C
PCB 166	EPA 1668A
	EPA 1668C
PCB 167	EPA 1668A
	EPA 1668C
PCB 168	EPA 1668A
	EPA 1668C
PCB 169	EPA 1668A
	EPA 1668C
PCB 17	EPA 1668A
	EPA 1668C
PCB 170	EPA 1668A
	EPA 1668C
PCB 171	EPA 1668A
	EPA 1668C
PCB 172	EPA 1668A
	EPA 1668C
PCB 173	EPA 1668A
	EPA 1668C
PCB 174	EPA 1668A
	EPA 1668C
PCB 175	EPA 1668A
	EPA 1668C
PCB 176	EPA 1668A
	EPA 1668C
PCB 177	EPA 1668A

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Polychlorinated Biphenyls

PCB 177	EPA 1668C
PCB 178	EPA 1668A
	EPA 1668C
PCB 179	EPA 1668A
	EPA 1668C
PCB 18	EPA 1668A
	EPA 1668C
PCB 180	EPA 1668A
	EPA 1668C
PCB 181	EPA 1668A
	EPA 1668C
PCB 182	EPA 1668A
	EPA 1668C
PCB 183	EPA 1668A
	EPA 1668C
PCB 184	EPA 1668A
	EPA 1668C
PCB 185	EPA 1668A
	EPA 1668C
PCB 186	EPA 1668A
	EPA 1668C
PCB 187	EPA 1668A
	EPA 1668C
PCB 188	EPA 1668A
	EPA 1668C
PCB 189	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 19	EPA 1668A
	EPA 1668C
PCB 190	EPA 1668A
	EPA 1668C
PCB 191	EPA 1668A
	EPA 1668C
PCB 192	EPA 1668A
	EPA 1668C
PCB 193	EPA 1668A
	EPA 1668C
PCB 194	EPA 1668A
	EPA 1668C
PCB 195	EPA 1668A
	EPA 1668C
PCB 196	EPA 1668A
	EPA 1668C
PCB 197	EPA 1668A
	EPA 1668C
PCB 198	EPA 1668A
	EPA 1668C
PCB 199	EPA 1668A
	EPA 1668C
PCB 2	EPA 1668A
	EPA 1668C
PCB 20	EPA 1668A
	EPA 1668C
PCB 200	EPA 1668A

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PCB 200	EPA 1668C
PCB 201	EPA 1668A
	EPA 1668C
PCB 202	EPA 1668A
	EPA 1668C
PCB 203	EPA 1668A
	EPA 1668C
PCB 204	EPA 1668A
	EPA 1668C
PCB 205	EPA 1668A
	EPA 1668C
PCB 206	EPA 1668A
	EPA 1668C
PCB 207	EPA 1668A
	EPA 1668C
PCB 208	EPA 1668A
	EPA 1668C
PCB 209	EPA 1668A
	EPA 1668C
PCB 21	EPA 1668A
	EPA 1668C
PCB 22	EPA 1668A
	EPA 1668C
PCB 23	EPA 1668A
	EPA 1668C
PCB 24	EPA 1668A
	EPA 1668C

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PCB 25	EPA 1668A
	EPA 1668C
PCB 26	EPA 1668A
	EPA 1668C
PCB 27	EPA 1668A
	EPA 1668C
PCB 28	EPA 1668A
	EPA 1668C
PCB 29	EPA 1668A
	EPA 1668C
PCB 3	EPA 1668A
	EPA 1668C
PCB 30	EPA 1668A
	EPA 1668C
PCB 31	EPA 1668A
	EPA 1668C
PCB 32	EPA 1668A
	EPA 1668C
PCB 33	EPA 1668A
	EPA 1668C
PCB 34	EPA 1668A
	EPA 1668C
PCB 35	EPA 1668A
	EPA 1668C
PCB 36	EPA 1668A
	EPA 1668C
PCB 37	EPA 1668A

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PCB 37	EPA 1668C
PCB 38	EPA 1668A
	EPA 1668C
PCB 39	EPA 1668A
	EPA 1668C
PCB 4	EPA 1668A
	EPA 1668C
PCB 40	EPA 1668A
	EPA 1668C
PCB 41	EPA 1668A
	EPA 1668C
PCB 42	EPA 1668A
	EPA 1668C
PCB 43	EPA 1668A
	EPA 1668C
PCB 44	EPA 1668A
	EPA 1668C
PCB 45	EPA 1668A
	EPA 1668C
PCB 46	EPA 1668A
	EPA 1668C
PCB 47	EPA 1668A
	EPA 1668C
PCB 48	EPA 1668A
	EPA 1668C
PCB 49	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 5	EPA 1668A
	EPA 1668C
PCB 50	EPA 1668A
	EPA 1668C
PCB 51	EPA 1668A
	EPA 1668C
PCB 52	EPA 1668A
	EPA 1668C
PCB 53	EPA 1668A
	EPA 1668C
PCB 54	EPA 1668A
	EPA 1668C
PCB 55	EPA 1668A
	EPA 1668C
PCB 56	EPA 1668A
	EPA 1668C
PCB 57	EPA 1668A
	EPA 1668C
PCB 58	EPA 1668A
	EPA 1668C
PCB 59	EPA 1668A
	EPA 1668C
PCB 6	EPA 1668A
	EPA 1668C
PCB 60	EPA 1668A
	EPA 1668C
PCB 61	EPA 1668A

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Polychlorinated Biphenyls

PCB 61	EPA 1668C
PCB 62	EPA 1668A
	EPA 1668C
PCB 63	EPA 1668A
	EPA 1668C
PCB 64	EPA 1668A
	EPA 1668C
PCB 65	EPA 1668A
	EPA 1668C
PCB 66	EPA 1668A
	EPA 1668C
PCB 67	EPA 1668A
	EPA 1668C
PCB 68	EPA 1668A
	EPA 1668C
PCB 69	EPA 1668A
	EPA 1668C
PCB 7	EPA 1668A
	EPA 1668C
PCB 70	EPA 1668A
	EPA 1668C
PCB 71	EPA 1668A
	EPA 1668C
PCB 72	EPA 1668A
	EPA 1668C
PCB 73	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 74	EPA 1668A
	EPA 1668C
PCB 75	EPA 1668A
	EPA 1668C
PCB 76	EPA 1668A
	EPA 1668C
PCB 77	EPA 1668A
	EPA 1668C
PCB 78	EPA 1668A
	EPA 1668C
PCB 79	EPA 1668A
	EPA 1668C
PCB 8	EPA 1668A
	EPA 1668C
PCB 80	EPA 1668A
	EPA 1668C
PCB 81	EPA 1668A
	EPA 1668C
PCB 82	EPA 1668A
	EPA 1668C
PCB 83	EPA 1668A
	EPA 1668C
PCB 84	EPA 1668A
	EPA 1668C
PCB 85	EPA 1668A
	EPA 1668C
PCB 86	EPA 1668A

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Polychlorinated Biphenyls

PCB 86	EPA 1668C
PCB 87	EPA 1668A
	EPA 1668C
PCB 88	EPA 1668A
	EPA 1668C
PCB 89	EPA 1668A
	EPA 1668C
PCB 9	EPA 1668A
	EPA 1668C
PCB 90	EPA 1668A
	EPA 1668C
PCB 91	EPA 1668A
	EPA 1668C
PCB 92	EPA 1668A
	EPA 1668C
PCB 93	EPA 1668A
	EPA 1668C
PCB 94	EPA 1668A
	EPA 1668C
PCB 95	EPA 1668A
	EPA 1668C
PCB 96	EPA 1668A
	EPA 1668C
PCB 97	EPA 1668A
	EPA 1668C
PCB 98	EPA 1668A
	EPA 1668C

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Polychlorinated Biphenyls

PCB 99	EPA 1668A
	EPA 1668C
PCB Congeners, Total	EPA 1668C

Polynuclear Aromatic Hydrocarbons

2-Acetylaminofluorene	EPA 8270E
3-Methylcholanthrene	EPA 8270E
7,12-Dimethylbenzyl (a) anthracene	EPA 8270E
Acenaphthene	EPA 8270E
Acenaphthylene	EPA 8270E
Anthracene	EPA 8270E
Benzo(a)anthracene	EPA 8270E
Benzo(a)pyrene	EPA 8270E
Benzo(b)fluoranthene	EPA 8270E
Benzo(g,h,i)perylene	EPA 8270E
Benzo(k)fluoranthene	EPA 8270E
Chrysene	EPA 8270E
Dibenzo(a,h)anthracene	EPA 8270E
Dibenzo(a,j)acridine	EPA 8270E
Fluoranthene	EPA 8270E
Fluorene	EPA 8270E
Indeno(1,2,3-cd)pyrene	EPA 8270E
Naphthalene	EPA 8270E
Phenanthrene	EPA 8270E
Pyrene	EPA 8270E

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270E

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Priority Pollutant Phenols

2,4,5-Trichlorophenol	EPA 8270E
2,4,6-Trichlorophenol	EPA 8270E
2,4-Dichlorophenol	EPA 8270E
2,4-Dimethylphenol	EPA 8270E
2,4-Dinitrophenol	EPA 8270E
2,6-Dichlorophenol	EPA 8270E
2-Chlorophenol	EPA 8270E
2-Methyl-4,6-dinitrophenol	EPA 8270E
2-Methylphenol	EPA 8270E
2-Nitrophenol	EPA 8270E
3-Methylphenol	EPA 8270E
4-Chloro-3-methylphenol	EPA 8270E
4-Methylphenol	EPA 8270E
4-Nitrophenol	EPA 8270E
Pentachlorophenol	EPA 8270E
Phenol	EPA 8270E

Semi-Volatile Organics

1,1'-Biphenyl	EPA 8270E
1,2-Dichlorobenzene, Semi-volatile	EPA 8270E
1,3-Dichlorobenzene, Semi-volatile	EPA 8270E
1,4-Dichlorobenzene, Semi-volatile	EPA 8270E
2-Methylnaphthalene	EPA 8270E
2-Picoline	EPA 8270E
4-Amino biphenyl	EPA 8270E
Acetophenone	EPA 8270E
Benzaldehyde	EPA 8270E
Benzoic Acid	EPA 8270E

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Semi-Volatile Organics

Benzyl alcohol	EPA 8270E
Caprolactam	EPA 8270E
Dibenzofuran	EPA 8270E
Ethyl methanesulfonate	EPA 8270E
Isosafrole	EPA 8270E
Methyl methanesulfonate	EPA 8270E
O,O,O-Triethyl phosphorothioate	EPA 8270E
Phenacetin	EPA 8270E
Safrole	EPA 8270E

Volatile Aromatics

1,2,4-Trichlorobenzene, Volatile	EPA 8260D
1,2,4-Trimethylbenzene	EPA 8260D
1,2-Dichlorobenzene	EPA 8260D
1,3,5-Trimethylbenzene	EPA 8260D
1,3-Dichlorobenzene	EPA 8260D
1,4-Dichlorobenzene	EPA 8260D
2-Chlorotoluene	EPA 8260D
4-Chlorotoluene	EPA 8260D
Benzene	EPA 8260D
Bromobenzene	EPA 8260D
Chlorobenzene	EPA 8260D
Ethyl benzene	EPA 8260D
Isopropylbenzene	EPA 8260D
m/p-Xylenes	EPA 8260D
Naphthalene, Volatile	EPA 8260D
n-Butylbenzene	EPA 8260D
n-Propylbenzene	EPA 8260D

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CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. DUANE LUCKENBILL *NY Lab Id No: 10670*
EUROFINS LANCASTER LABORATORIES ENVIRONMENT
TESTING, LLC
2425 NEW HOLLAND PIKE
LANCASTER, PA 17601-5994

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Volatile Aromatics

o-Xylene	EPA 8260D
p-Isopropyltoluene (P-Cymene)	EPA 8260D
sec-Butylbenzene	EPA 8260D
Styrene	EPA 8260D
tert-Butylbenzene	EPA 8260D
Toluene	EPA 8260D
Total Xylenes	EPA 8260D

Volatile Chlorinated Organics

Benzyl chloride	EPA 8260D

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 8260D
1,1,1-Trichloroethane	EPA 8260D
1,1,2,2-Tetrachloroethane	EPA 8260D
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260D
1,1,2-Trichloroethane	EPA 8260D
1,1-Dichloroethane	EPA 8260D
1,1-Dichloroethene	EPA 8260D
1,1-Dichloropropene	EPA 8260D
1,2,3-Trichloropropane	EPA 8260D
1,2-Dibromo-3-chloropropane	EPA 8260D
1,2-Dibromoethane	EPA 8260D
1,2-Dichloroethane	EPA 8260D
1,2-Dichloropropane	EPA 8260D
1,3-Dichloropropane	EPA 8260D
2,2-Dichloropropane	EPA 8260D
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260D

Serial No.: 68525

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

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Volatile Halocarbons

2-Chloroethylvinyl ether	EPA 8260D
3-Chloropropene (Allyl chloride)	EPA 8260D
Bromochloromethane	EPA 8260D
Bromodichloromethane	EPA 8260D
Bromoform	EPA 8260D
Bromomethane	EPA 8260D
Carbon tetrachloride	EPA 8260D
Chloroethane	EPA 8260D
Chloroform	EPA 8260D
Chloromethane	EPA 8260D
cis-1,2-Dichloroethene	EPA 8260D
cis-1,3-Dichloropropene	EPA 8260D
Dibromochloromethane	EPA 8260D
Dibromomethane	EPA 8260D
Dichlorodifluoromethane	EPA 8260D
Hexachlorobutadiene, Volatile	EPA 8260D
Methyl iodide	EPA 8260D
Methylene chloride	EPA 8260D
Tetrachloroethene	EPA 8260D
trans-1,2-Dichloroethene	EPA 8260D
trans-1,3-Dichloropropene	EPA 8260D
trans-1,4-Dichloro-2-butene	EPA 8260D
Trichloroethene	EPA 8260D
Trichlorofluoromethane	EPA 8260D
Vinyl chloride	EPA 8260D

Volatile Organics

1,4-Dioxane	EPA 8260D

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Volatile Organics

1,4-Dioxane	EPA 8260C SIM
	EPA 8270E
	EPA 8270E SIM
2-Butanone (Methylethyl ketone)	EPA 8260D
2-Hexanone	EPA 8260D
2-Nitropropane	EPA 8260D
4-Methyl-2-Pentanone	EPA 8260D
Acetone	EPA 8260D
Acetonitrile	EPA 8260D
Carbon Disulfide	EPA 8260D
Cyclohexane	EPA 8260D
Ethyl Acetate	EPA 8260D
Ethylene Glycol	EPA 8015D
	EPA 8015C
Isobutyl alcohol	EPA 8260D
Isopropanol	EPA 8260D
Methyl acetate	EPA 8260D
Methyl cyclohexane	EPA 8260D
Methyl tert-butyl ether	EPA 8260D
n-Butanol	EPA 8260D
o-Toluidine	EPA 8270E
Propionitrile	EPA 8260D
tert-butyl alcohol	EPA 8260D
Tetrahydrofuran	EPA 8260D
Vinyl acetate	EPA 8260D

Sample Preparation Methods

	EPA 5035A-L

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Sample Preparation Methods

EPA 5035A-H
EPA 3580A
EPA 3010A
EPA 3005A
EPA 3050B
EPA 3550C
EPA 3540C
EPA 3546
EPA 5035
EPA 3060A

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ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved subcategories and/or analytes are listed below:

Chlorophenoxy Acid Pesticides

Dinoseb	EPA 8270E

Nitroaromatics and Isophorone

1,3,5-Trinitrobenzene	EPA 8270E

Perfluorinated Alkyl Acids

8:2FTS	EPA 1633 (Draft)
NETFOSAA	EPA 1633 (Draft)
NMEFOSAA	EPA 1633 (Draft)
Perflourotridecanoic Acid (PFTRDA)	EPA 1633 (Draft)
Perfluordecanoic Acid (PFDA)	EPA 1633 (Draft)
Perfluorobutanoic Acid (PFBA)	EPA 1633 (Draft)
Perfluorododecanoic Acid (PFDOA)	EPA 1633 (Draft)
Perfluoroheptanoic Acid (PFHPA)	EPA 1633 (Draft)
Perfluorohexanoic Acid (PFHXA)	EPA 1633 (Draft)
Perfluorononanoic Acid (PFNA)	EPA 1633 (Draft)
Perfluorooctanesulfonic Acid (PFOS)	EPA 1633 (Draft)
Perfluorooctanoic Acid (PFOA)	EPA 1633 (Draft)
Perfluoropentanoic Acid (PFPEA)	EPA 1633 (Draft)
Perfluorotetradecanoic Acid (PFTA)	EPA 1633 (Draft)
Perfluoroundecanoic Acid (PFUNA)	EPA 1633 (Draft)

Sample Preparation Methods

	EPA 3050B
	EPA 3546

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All approved analytes are listed below:

Acrylates

Acetonitrile	EPA TO-15
Acrylonitrile	EPA TO-15
Ethyl acrylate	EPA TO-15
Methyl methacrylate	EPA TO-15

Chlorinated Hydrocarbons

1,2,4-Trichlorobenzene	EPA TO-15
Hexachlorobutadiene	EPA TO-15
Hexachloroethane	EPA TO-15

Polynuclear Aromatics

Naphthalene	EPA TO-15

Purgeable Aromatics

1,2,4-Trimethylbenzene	EPA TO-15
1,2-Dichlorobenzene	EPA TO-15
1,3,5-Trimethylbenzene	EPA TO-15
1,3-Dichlorobenzene	EPA TO-15
1,4-Dichlorobenzene	EPA TO-15
2-Chlorotoluene	EPA TO-15
Benzene	EPA TO-15
Chlorobenzene	EPA TO-15
Ethyl benzene	EPA TO-15
Isopropylbenzene	EPA TO-15
m/p-Xylenes	EPA TO-15
o-Xylene	EPA TO-15
Styrene	EPA TO-15
Toluene	EPA TO-15
Total Xylenes	EPA TO-15

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All approved analytes are listed below:

Purgeable Halocarbons

1,1,1-Trichloroethane	EPA TO-15
1,1,2,2-Tetrachloroethane	EPA TO-15
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA TO-15
1,1,2-Trichloroethane	EPA TO-15
1,1-Dichloroethane	EPA TO-15
1,1-Dichloroethene	EPA TO-15
1,2-Dibromo-3-chloropropane	EPA TO-15
1,2-Dibromoethane	EPA TO-15
1,2-Dichloroethane	EPA TO-15
1,2-Dichloropropane	EPA TO-15
3-Chloropropene (Allyl chloride)	EPA TO-15
Bromodichloromethane	EPA TO-15
Bromoform	EPA TO-15
Bromomethane	EPA TO-15
Carbon tetrachloride	EPA TO-15
Chloroethane	EPA TO-15
Chloroform	EPA TO-15
Chloromethane	EPA TO-15
cis-1,2-Dichloroethene	EPA TO-15
cis-1,3-Dichloropropene	EPA TO-15
Dibromochloromethane	EPA TO-15
Dichlorodifluoromethane	EPA TO-15
Methylene chloride	EPA TO-15
Tetrachloroethene	EPA TO-15
trans-1,2-Dichloroethene	EPA TO-15
trans-1,3-Dichloropropene	EPA TO-15
Trichloroethene	EPA TO-15

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Purgeable Halocarbons

Trichlorofluoromethane	EPA TO-15
Vinyl bromide	EPA TO-15
Vinyl chloride	EPA TO-15

Volatile Chlorinated Organics

Benzyl chloride	EPA TO-15

Volatile Organics

1,2-Dichlorotetrafluoroethane	EPA TO-15
1,3-Butadiene	EPA TO-15
1,4-Dioxane	EPA TO-15
2,2,4-Trimethylpentane	EPA TO-15
2-Butanone (Methylethyl ketone)	EPA TO-15
4-Methyl-2-Pentanone	EPA TO-15
Acetone	EPA TO-15
Acrolein (Propenal)	EPA TO-15
Carbon Disulfide	EPA TO-15
Cyclohexane	EPA TO-15
Hexane	EPA TO-15
Isopropanol	EPA TO-15
Methyl iodide	EPA TO-15
Methyl tert-butyl ether	EPA TO-15
n-Heptane	EPA TO-15
tert-butyl alcohol	EPA TO-15
Vinyl acetate	EPA TO-15

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MS. CATALINA DALANGIN
EUROFINS EDISON
777 NEW DURHAM ROAD
EDISON, NJ 08817

NY Lab Id No: 11452

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Fuel Additives

Methyl tert-butyl ether	EPA 524.2
Naphthalene	EPA 524.2

Metals I

Arsenic, Total	EPA 200.8 Rev. 5.4
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Iron, Total	EPA 200.7 Rev. 4.4
Lead, Total	EPA 200.8 Rev. 5.4
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Mercury, Total	EPA 245.1 Rev. 3.0
Selenium, Total	EPA 200.8 Rev. 5.4
Silver, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4

Metals II

Aluminum, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Antimony, Total	EPA 200.8 Rev. 5.4

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All approved analytes are listed below:

Metals II

Beryllium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Nickel, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Thallium, Total	EPA 200.8 Rev. 5.4
Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4

Metals III

Boron, Total	EPA 200.7 Rev. 4.4
Calcium, Total	EPA 200.7 Rev. 4.4
Magnesium, Total	EPA 200.7 Rev. 4.4
Potassium, Total	EPA 200.7 Rev. 4.4
Sodium, Total	EPA 200.7 Rev. 4.4

Miscellaneous

Methyl iodide	EPA 524.2
Organic Carbon, Dissolved	SM 21-23 5310B (-00)
Organic Carbon, Total	SM 21-23 5310B (-00)
Turbidity	SM 21-23 2130 B (-01)
	EPA 180.1 Rev. 2.0

Non-Metals

Calcium Hardness	EPA 200.7 Rev. 4.4
	SM 18-22 2340B (-97)
Chloride	SM 21-23 4500-Cl- B (-97)
	EPA 300.0 Rev. 2.1
Color	SM 21-23 2120B (-01)
Cyanide	LACHAT QuikChem 10-204-00-1-X

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Non-Metals

Cyanide	EPA 335.4 Rev. 1.0
Fluoride, Total	EPA 300.0 Rev. 2.1
	SM 21-23 4500-F C (-97)
Nitrate (as N)	EPA 353.2 Rev. 2.0
	EPA 300.0 Rev. 2.1
	SM 21-23 4500-NO3 F (-00)
Nitrite (as N)	EPA 353.2 Rev. 2.0
	EPA 300.0 Rev. 2.1
	SM 21-23 4500-NO3 F (-00)
Orthophosphate (as P)	SM 19, 21-23 4500-P E (-99)
Solids, Total Dissolved	SM 21-23 2540C (-97)
Specific Conductance	SM 21-23 2510B (-97)
Sulfate (as SO4)	EPA 300.0 Rev. 2.1

Trihalomethanes

Bromodichloromethane	EPA 524.2
Bromoform	EPA 524.2
Chloroform	EPA 524.2
Dibromochloromethane	EPA 524.2

Volatile Aromatics

1,2,3-Trichlorobenzene	EPA 524.2
1,2,4-Trichlorobenzene	EPA 524.2
1,2,4-Trimethylbenzene	EPA 524.2
1,2-Dichlorobenzene	EPA 524.2
1,3,5-Trimethylbenzene	EPA 524.2
1,3-Dichlorobenzene	EPA 524.2
1,4-Dichlorobenzene	EPA 524.2

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Volatile Aromatics

2-Chlorotoluene	EPA 524.2
4-Chlorotoluene	EPA 524.2
Benzene	EPA 524.2
Bromobenzene	EPA 524.2
Chlorobenzene	EPA 524.2
Ethyl benzene	EPA 524.2
Hexachlorobutadiene	EPA 524.2
Isopropylbenzene	EPA 524.2
n-Butylbenzene	EPA 524.2
n-Propylbenzene	EPA 524.2
p-Isopropyltoluene (P-Cymene)	EPA 524.2
sec-Butylbenzene	EPA 524.2
Styrene	EPA 524.2
tert-Butylbenzene	EPA 524.2
Toluene	EPA 524.2
Total Xylenes	EPA 524.2

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2
1,1-Dichloropropene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2
1,2-Dichloroethane	EPA 524.2
1,2-Dichloropropane	EPA 524.2

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All approved analytes are listed below:

Volatile Halocarbons

1,3-Dichloropropane	EPA 524.2
2,2-Dichloropropane	EPA 524.2
Bromochloromethane	EPA 524.2
Bromomethane	EPA 524.2
Carbon tetrachloride	EPA 524.2
Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2
Methylene chloride	EPA 524.2
Tetrachloroethene	EPA 524.2
trans-1,2-Dichloroethene	EPA 524.2
trans-1,3-Dichloropropene	EPA 524.2
Trichloroethene	EPA 524.2
Trichlorofluoromethane	EPA 524.2
Vinyl chloride	EPA 524.2

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WADSWORTH CENTER

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EUROFINS EDISON
777 NEW DURHAM ROAD
EDISON, NJ 08817

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All approved analytes are listed below:

Acrylates

Acrolein (Propenal)	EPA 8260D
	EPA 624.1
Acrylonitrile	EPA 8260D
	EPA 624.1
Ethyl methacrylate	EPA 8260D
Methyl methacrylate	EPA 8260D

Amines

1,2-Diphenylhydrazine	EPA 625.1
	EPA 8270E
1-Naphthylamine	EPA 8270E
2,3-Dichloroaniline	EPA 625.1
2-Naphthylamine	EPA 8270E
2-Nitroaniline	EPA 8270E
3-Nitroaniline	EPA 8270E
4-Chloroaniline	EPA 8270E
4-Nitroaniline	EPA 8270E
Aniline	EPA 625.1
	EPA 8270E
Carbazole	EPA 625.1
	EPA 8270E
Diphenylamine	EPA 8270E
Propionitrile	EPA 8260D
Pyridine	EPA 625.1
	EPA 8270E

Benzidines

3,3'-Dichlorobenzidine	EPA 625.1

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Benzidines

3,3'-Dichlorobenzidine	EPA 8270E
Benzidine	EPA 625.1
	EPA 8270E

Chlorinated Hydrocarbon Pesticides

4,4'-DDD	EPA 8081B
	EPA 608.3
4,4'-DDE	EPA 8081B
	EPA 608.3
4,4'-DDT	EPA 8081B
	EPA 608.3
Aldrin	EPA 8081B
	EPA 608.3
alpha-BHC	EPA 8081B
	EPA 608.3
alpha-Chlordane	EPA 8081B
	EPA 608.3
beta-BHC	EPA 8081B
	EPA 608.3
Chlordane Total	EPA 8081B
	EPA 608.3
delta-BHC	EPA 8081B
	EPA 608.3
Dieldrin	EPA 8081B
	EPA 608.3
Endosulfan I	EPA 8081B
	EPA 608.3
Endosulfan II	EPA 8081B

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Chlorinated Hydrocarbon Pesticides

Endosulfan II	EPA 608.3
Endosulfan sulfate	EPA 8081B
	EPA 608.3
Endrin	EPA 8081B
	EPA 608.3
Endrin aldehyde	EPA 8081B
	EPA 608.3
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
	EPA 608.3
Heptachlor	EPA 8081B
	EPA 608.3
Heptachlor epoxide	EPA 8081B
	EPA 608.3
Lindane	EPA 8081B
	EPA 608.3
Methoxychlor	EPA 8081B
	EPA 608.3
Mirex	EPA 8081B
Toxaphene	EPA 8081B
	EPA 608.3

Chlorinated Hydrocarbons

1,2,3-Trichlorobenzene	EPA 8260D
1,2,4,5-Tetrachlorobenzene	EPA 8270E
1,2,4-Trichlorobenzene	EPA 625.1
	EPA 8270E
2-Chloronaphthalene	EPA 625.1

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Chlorinated Hydrocarbons

2-Chloronaphthalene	EPA 8270E
Hexachlorobenzene	EPA 625.1
	EPA 8270E
Hexachlorobutadiene	EPA 625.1
	EPA 8270E
Hexachlorocyclopentadiene	EPA 625.1
	EPA 8270E
Hexachloroethane	EPA 625.1
	EPA 8270E

Chlorophenoxy Acid Pesticides

2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
Pentachlorophenol	EPA 8151A

Demand

Biochemical Oxygen Demand	SM 5210B-2016
Carbonaceous BOD	SM 5210B-2016
Chemical Oxygen Demand	EPA 410.4, Rev. 2.0 (1993)
	SM 5220D-2011

Fuel Oxygenates

Di-isopropyl ether	EPA 8260D

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Fuel Oxygenates

Ethanol	EPA 8260D
	EPA 624.1
Methyl tert-butyl ether	EPA 8260D
	EPA 624.1
tert-amyl methyl ether (TAME)	EPA 8260D
tert-butyl alcohol	EPA 8260D
	EPA 624.1
tert-butyl ethyl ether (ETBE)	EPA 8260D

Haloethers

2,2'-Oxybis(1-chloropropane)	EPA 625.1
	EPA 8270E
4-Bromophenylphenyl ether	EPA 625.1
	EPA 8270E
4-Chlorophenylphenyl ether	EPA 625.1
	EPA 8270E
Bis(2-chloroethoxy)methane	EPA 625.1
	EPA 8270E
Bis(2-chloroethyl)ether	EPA 625.1
	EPA 8270E

Low Level Polynuclear Aromatics

Acenaphthene Low Level	EPA 8270E SIM
Acenaphthylene Low Level	EPA 8270E SIM
Anthracene Low Level	EPA 8270E SIM
Benzo(a)anthracene Low Level	EPA 8270E SIM
Benzo(a)pyrene Low Level	EPA 8270E SIM
Benzo(b)fluoranthene Low Level	EPA 8270E SIM

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Low Level Polynuclear Aromatics

Benzo(g,h,i)perylene Low Level	EPA 8270E SIM
Benzo(k)fluoranthene Low Level	EPA 8270E SIM
Chrysene Low Level	EPA 8270E SIM
Dibenzo(a,h)anthracene Low Level	EPA 8270E SIM
Fluoranthene Low Level	EPA 8270E SIM
Fluorene Low Level	EPA 8270E SIM
Indeno(1,2,3-cd)pyrene Low Level	EPA 8270E SIM
Naphthalene Low Level	EPA 8270E SIM
Phenanthrene Low Level	EPA 8270E SIM
Pyrene Low Level	EPA 8270E SIM

Metals I

Barium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Cadmium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Calcium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Chromium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)

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Metals I

Copper, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Iron, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Lead, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Magnesium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Manganese, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Nickel, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Potassium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B

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Metals I

Potassium, Total	EPA 200.8, Rev. 5.4 (1994)
Silver, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Sodium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Strontium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B

Metals II

Aluminum, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Antimony, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Arsenic, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Beryllium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D

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Metals II

Beryllium, Total	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Chromium VI	USGS I-1230-85
	EPA 7196A
Mercury, Total	EPA 245.1, Rev. 3.0 (1994)
	EPA 7470A
Selenium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Vanadium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Zinc, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)

Metals III

Cobalt, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Molybdenum, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)

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Metals III

Thallium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Tin, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Titanium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)

Mineral

Acidity	SM 2310B-2011
Alkalinity	SM 2320B-2011
Chloride	SM 4500-Cl- B-2011
	EPA 300.0, Rev. 2.1 (1993)
	SM 4500-Cl- E-2011
	EPA 9253
Fluoride, Total	EPA 300.0, Rev. 2.1 (1993)
Hardness, Total	SM 2340C-2011
	SM 2340B-2011
Sulfate (as SO4)	ASTM D516-16
	EPA 300.0, Rev. 2.1 (1993)

Miscellaneous

Boron, Total	EPA 200.7, Rev. 4.4 (1994)

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Miscellaneous

Boron, Total	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Bromide	EPA 300.0, Rev. 2.1 (1993)
Color	SM 2120B-2011
Cyanide, Total	LACHAT QuikChem 10-204-00-1-X
	EPA 335.4, Rev. 1.0 (1993)
	EPA 9012B
Oil and Grease Total Recoverable	EPA 1664A
Organic Carbon, Total	SM 5310B-2014
	EPA 9060A
Phenols	EPA 420.1 (Rev. 1978)
	EPA 9065
Specific Conductance	EPA 120.1 (Rev. 1982)
	SM 2510B-2011
	EPA 9050A
Sulfide (as S)	SM 4500-S2- F-2011
	EPA 9034
Turbidity	SM 2130 B-2011
	EPA 180.1, Rev. 2.0 (1993)

Nitroaromatics and Isophorone

2,4-Dinitrotoluene	EPA 625.1
	EPA 8270E
2,6-Dinitrotoluene	EPA 625.1
	EPA 8270E
Isophorone	EPA 625.1
	EPA 8270E

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Nitroaromatics and Isophorone	
Nitrobenzene	EPA 625.1
	EPA 8270E
Nitrosoamines	
N-Nitrosodimethylamine	EPA 625.1
	EPA 8270E
N-Nitrosodi-n-propylamine	EPA 625.1
	EPA 8270E
N-Nitrosodiphenylamine	EPA 625.1
	EPA 8270E
Nutrient	
Ammonia (as N)	SM 4500-NH3 H-2011
	EPA 350.1, Rev. 2.0 (1993)
Nitrate (as N)	EPA 300.0, Rev. 2.1 (1993)
Nitrate-Nitrite (as N)	EPA 353.2, Rev. 2.0 (1993)
	EPA 300.0, Rev. 2.1 (1993)
	SM 4500-NO3 F-2016
Nitrite (as N)	EPA 353.2, Rev. 2.0 (1993)
	EPA 300.0, Rev. 2.1 (1993)
	SM 4500-NO3 F-2016
Orthophosphate (as P)	SM 4500-P E-2011
Phosphorus, Total	SM 4500-P E-2011
Organophosphate Pesticides	
Atrazine	EPA 8270E
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015D
Gasoline Range Organics	EPA 8015D

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WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. CATALINA DALANGIN
EUROFINS EDISON
777 NEW DURHAM ROAD
EDISON, NJ 08817

NY Lab Id No: 11452

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Phthalate Esters

Benzyl butyl phthalate	EPA 625.1
	EPA 8270E
Bis(2-ethylhexyl) phthalate	EPA 625.1
	EPA 8270E
Diethyl phthalate	EPA 625.1
	EPA 8270E
Dimethyl phthalate	EPA 625.1
	EPA 8270E
Di-n-butyl phthalate	EPA 625.1
	EPA 8270E
Di-n-octyl phthalate	EPA 625.1
	EPA 8270E

Polychlorinated Biphenyls

Aroclor 1016 (PCB-1016)	EPA 8082A
	EPA 608.3
Aroclor 1221 (PCB-1221)	EPA 8082A
	EPA 608.3
Aroclor 1232 (PCB-1232)	EPA 8082A
	EPA 608.3
Aroclor 1242 (PCB-1242)	EPA 8082A
	EPA 608.3
Aroclor 1248 (PCB-1248)	EPA 8082A
	EPA 608.3
Aroclor 1254 (PCB-1254)	EPA 8082A
	EPA 608.3
Aroclor 1260 (PCB-1260)	EPA 8082A
	EPA 608.3

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Polychlorinated Biphenyls

Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A

Polynuclear Aromatics

Acenaphthene	EPA 625.1
	EPA 8270E
Acenaphthylene	EPA 625.1
	EPA 8270E
Anthracene	EPA 625.1
	EPA 8270E
Benzo(a)anthracene	EPA 625.1
	EPA 8270E
Benzo(a)pyrene	EPA 625.1
	EPA 8270E
Benzo(b)fluoranthene	EPA 625.1
	EPA 8270E
Benzo(g,h,i)perylene	EPA 625.1
	EPA 8270E
Benzo(k)fluoranthene	EPA 625.1
	EPA 8270E
Chrysene	EPA 625.1
	EPA 8270E
Dibenzo(a,h)anthracene	EPA 625.1
	EPA 8270E
Fluoranthene	EPA 625.1
	EPA 8270E
Fluorene	EPA 625.1
	EPA 8270E

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Polynuclear Aromatics

Indeno(1,2,3-cd)pyrene	EPA 625.1
	EPA 8270E
Naphthalene	EPA 625.1
	EPA 8270E
Phenanthrene	EPA 625.1
	EPA 8270E
Pyrene	EPA 625.1
	EPA 8270E

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270E
2,4,5-Trichlorophenol	EPA 625.1
	EPA 8270E
2,4,6-Trichlorophenol	EPA 625.1
	EPA 8270E
2,4-Dichlorophenol	EPA 625.1
	EPA 8270E
2,4-Dimethylphenol	EPA 625.1
	EPA 8270E
2,4-Dinitrophenol	EPA 625.1
	EPA 8270E
2-Chlorophenol	EPA 625.1
	EPA 8270E
2-Methyl-4,6-dinitrophenol	EPA 625.1
	EPA 8270E
2-Methylphenol	EPA 625.1
	EPA 8270E
2-Nitrophenol	EPA 625.1

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Priority Pollutant Phenols

2-Nitrophenol	EPA 8270E
3-Methylphenol	EPA 625.1
	EPA 8270E
4-Chloro-3-methylphenol	EPA 625.1
	EPA 8270E
4-Methylphenol	EPA 625.1
	EPA 8270E
4-Nitrophenol	EPA 625.1
	EPA 8270E
Cresols, Total	EPA 8270E
Pentachlorophenol	EPA 625.1
	EPA 8270E
Phenol	EPA 625.1
	EPA 8270E

Residue

Settleable Solids	SM 2540 F-2015
Solids, Total	SM 2540 B-2015
Solids, Total Dissolved	SM 2540 C-2015
Solids, Total Suspended	SM 2540 D-2015
Solids, Volatile	EPA 160.4 (Issued 1971)

Semi-Volatile Organics

1,1'-Biphenyl	EPA 8270E
1,2-Dichlorobenzene, Semi-volatile	EPA 8270E
1,3-Dichlorobenzene, Semi-volatile	EPA 8270E
1,4-Dichlorobenzene, Semi-volatile	EPA 8270E
2-Methylnaphthalene	EPA 625.1

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Semi-Volatile Organics

2-Methylnaphthalene	EPA 8270E
Acetophenone	EPA 625.1
	EPA 8270E
alpha-Terpineol	EPA 625.1
Benzaldehyde	EPA 8270E
Benzoic Acid	EPA 8270E
Benzyl alcohol	EPA 8270E
Caprolactam	EPA 8270E
Dibenzofuran	EPA 8270E
n-Decane	EPA 625.1
	EPA 8270E
n-Octadecane	EPA 625.1
	EPA 8270E

Volatile Aromatics

1,2,4-Trichlorobenzene, Volatile	EPA 8260D
1,2,4-Trimethylbenzene	EPA 8260D
1,2-Dichlorobenzene	EPA 8260D
	EPA 624.1
1,3,5-Trimethylbenzene	EPA 8260D
1,3-Dichlorobenzene	EPA 8260D
	EPA 624.1
1,4-Dichlorobenzene	EPA 8260D
	EPA 624.1
2-Chlorotoluene	EPA 8260D
4-Chlorotoluene	EPA 8260D
Benzene	EPA 8260D
	EPA 624.1

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Volatile Aromatics

Bromobenzene	EPA 8260D
Chlorobenzene	EPA 8260D
	EPA 624.1
Ethyl benzene	EPA 8260D
	EPA 624.1
Isopropylbenzene	EPA 8260D
m/p-Xylenes	EPA 8260D
	EPA 624.1
Naphthalene, Volatile	EPA 8260D
n-Butylbenzene	EPA 8260D
n-Propylbenzene	EPA 8260D
o-Xylene	EPA 8260D
	EPA 624.1
p-Isopropyltoluene (P-Cymene)	EPA 8260D
sec-Butylbenzene	EPA 8260D
Styrene	EPA 8260D
	EPA 624.1
tert-Butylbenzene	EPA 8260D
Toluene	EPA 8260D
	EPA 624.1
Total Xylenes	EPA 8260D
	EPA 624.1

Volatile Chlorinated Organics

Benzyl chloride	EPA 8260D

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 8260D

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Volatile Halocarbons

1,1,1-Trichloroethane	EPA 8260D
	EPA 624.1
1,1,2,2-Tetrachloroethane	EPA 8260D
	EPA 624.1
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260D
	EPA 624.1
1,1,2-Trichloroethane	EPA 8260D
	EPA 624.1
1,1-Dichloroethane	EPA 8260D
	EPA 624.1
1,1-Dichloroethene	EPA 8260D
	EPA 624.1
1,1-Dichloropropene	EPA 8260D
1,2,3-Trichloropropane	EPA 8260D
1,2-Dibromo-3-chloropropane	EPA 8260D
1,2-Dibromoethane	EPA 8260D
1,2-Dichloroethane	EPA 8260D
	EPA 624.1
1,2-Dichloropropane	EPA 8260D
	EPA 624.1
1,3-Dichloropropane	EPA 8260D
2,2-Dichloropropane	EPA 8260D
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260D
2-Chloroethylvinyl ether	EPA 8260D
	EPA 624.1
3-Chloropropene (Allyl chloride)	EPA 8260D
Bromochloromethane	EPA 8260D

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Volatile Halocarbons

Bromodichloromethane	EPA 8260D
	EPA 624.1
Bromoform	EPA 8260D
	EPA 624.1
Bromomethane	EPA 8260D
	EPA 624.1
Carbon tetrachloride	EPA 8260D
	EPA 624.1
Chloroethane	EPA 8260D
	EPA 624.1
Chloroform	EPA 8260D
	EPA 624.1
Chloromethane	EPA 8260D
	EPA 624.1
cis-1,2-Dichloroethene	EPA 8260D
	EPA 624.1
cis-1,3-Dichloropropene	EPA 8260D
	EPA 624.1
Dibromochloromethane	EPA 8260D
	EPA 624.1
Dibromomethane	EPA 8260D
Dichlorodifluoromethane	EPA 8260D
	EPA 624.1
Hexachlorobutadiene, Volatile	EPA 8260D
Methyl iodide	EPA 8260D
Methylene chloride	EPA 8260D
	EPA 624.1

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Volatile Halocarbons

Tetrachloroethene	EPA 8260D
	EPA 624.1
trans-1,2-Dichloroethene	EPA 8260D
	EPA 624.1
trans-1,3-Dichloropropene	EPA 8260D
	EPA 624.1
trans-1,4-Dichloro-2-butene	EPA 8260D
Trichloroethene	EPA 8260D
	EPA 624.1
Trichlorofluoromethane	EPA 8260D
	EPA 624.1
Vinyl chloride	EPA 8260D
	EPA 624.1

Volatiles Organics

1,4-Dioxane	EPA 8260D
	EPA 8260D SIM
	EPA 8270E
	EPA 8270E SIM
2-Butanone (Methylethyl ketone)	EPA 8260D
2-Hexanone	EPA 8260D
2-Nitropropane	EPA 8260D
4-Methyl-2-Pentanone	EPA 8260D
	EPA 624.1
Acetone	EPA 8260D
	EPA 624.1
Acetonitrile	EPA 8260D
	EPA 624.1

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Volatiles Organics

Carbon Disulfide	EPA 8260D
Cyclohexane	EPA 8260D
Di-ethyl ether	EPA 8260D
Ethyl Acetate	EPA 8260D
Isobutyl alcohol	EPA 8260D
Isopropanol	EPA 8260D
Methyl acetate	EPA 8260D
Methyl cyclohexane	EPA 8260D
n-Butanol	EPA 8260D
o-Toluidine	EPA 8260D
Tetrahydrofuran	EPA 8260D
	EPA 624.1
Vinyl acetate	EPA 8260D
	EPA 624.1

Sample Preparation Methods

SM 4500-P B(5)-2011
EPA 5030C
SM 4500-CN B-2016 and C-2016
EPA 3511
EPA 9030B
EPA 3010A
EPA 3005A
EPA 3510C
EPA 3520C
SM 4500-NH3 B-2011

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All approved analytes are listed below:

Acrylates

Acrolein (Propenal)	EPA 8260D
Acrylonitrile	EPA 8260D
Ethyl methacrylate	EPA 8260D
Methyl acrylonitrile	EPA 8260D
Methyl methacrylate	EPA 8260D

Amines

1,2-Diphenylhydrazine	EPA 8270E
1-Naphthylamine	EPA 8270E
2-Naphthylamine	EPA 8270E
2-Nitroaniline	EPA 8270E
3-Nitroaniline	EPA 8270E
4-Chloroaniline	EPA 8270E
4-Nitroaniline	EPA 8270E
Aniline	EPA 8270E
Carbazole	EPA 8270E

Benzidines

3,3'-Dichlorobenzidine	EPA 8270E
Benzidine	EPA 8270E

Characteristic Testing

Corrosivity (pH)	EPA 9040C
	EPA 9045D
Free Liquids	EPA 9095B
Ignitability	EPA 1030
	EPA 1020C
Synthetic Precipitation Leaching Proc.	EPA 1312
TCLP	EPA 1311

Serial No.: 68780

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All approved analytes are listed below:

Chlorinated Hydrocarbon Pesticides

4,4'-DDD	EPA 8081B
4,4'-DDE	EPA 8081B
4,4'-DDT	EPA 8081B
Aldrin	EPA 8081B
alpha-BHC	EPA 8081B
alpha-Chlordane	EPA 8081B
Atrazine	EPA 8270E
beta-BHC	EPA 8081B
Chlordane Total	EPA 8081B
delta-BHC	EPA 8081B
Dieldrin	EPA 8081B
Endosulfan I	EPA 8081B
Endosulfan II	EPA 8081B
Endosulfan sulfate	EPA 8081B
Endrin	EPA 8081B
Endrin aldehyde	EPA 8081B
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B
Heptachlor epoxide	EPA 8081B
Lindane	EPA 8081B
Methoxychlor	EPA 8081B
Mirex	EPA 8081B
Pentachloronitrobenzene	EPA 8270E
Toxaphene	EPA 8081B

Chlorinated Hydrocarbons

1,2,3-Trichlorobenzene	EPA 8260D

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Chlorinated Hydrocarbons

1,2,4,5-Tetrachlorobenzene	EPA 8270E
1,2,4-Trichlorobenzene	EPA 8270E
2-Chloronaphthalene	EPA 8270E
Hexachlorobenzene	EPA 8270E
Hexachlorobutadiene	EPA 8270E
Hexachlorocyclopentadiene	EPA 8270E
Hexachloroethane	EPA 8270E

Chlorophenoxy Acid Pesticides

2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
MCPA	EPA 8151A
MCPP	EPA 8151A
Pentachlorophenol	EPA 8151A

Haloethers

2,2'-Oxybis(1-chloropropane)	EPA 8270E
4-Bromophenylphenyl ether	EPA 8270E
4-Chlorophenylphenyl ether	EPA 8270E
Bis(2-chloroethoxy)methane	EPA 8270E
Bis(2-chloroethyl)ether	EPA 8270E

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WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

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Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MS. CATALINA DALANGIN
EUROFINS EDISON
777 NEW DURHAM ROAD
EDISON, NJ 08817

NY Lab Id No: 11452

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All approved analytes are listed below:

Low Level Polynuclear Aromatic Hydrocarbons

Acenaphthene Low Level	EPA 8270E SIM
Acenaphthylene Low Level	EPA 8270E SIM
Anthracene Low Level	EPA 8270E SIM
Benzo(a)anthracene Low Level	EPA 8270E SIM
Benzo(a)pyrene Low Level	EPA 8270E SIM
Benzo(b)fluoranthene Low Level	EPA 8270E SIM
Benzo(g,h,i)perylene Low Level	EPA 8270E SIM
Benzo(k)fluoranthene Low Level	EPA 8270E SIM
Chrysene Low Level	EPA 8270E SIM
Dibenzo(a,h)anthracene Low Level	EPA 8270E SIM
Fluoranthene Low Level	EPA 8270E SIM
Fluorene Low Level	EPA 8270E SIM
Indeno(1,2,3-cd)pyrene Low Level	EPA 8270E SIM
Phenanthrene Low Level	EPA 8270E SIM
Pyrene Low Level	EPA 8270E SIM

Metals I

Barium, Total	EPA 6010D
	EPA 6020B
Cadmium, Total	EPA 6010D
	EPA 6020B
Calcium, Total	EPA 6010D
	EPA 6020B
Chromium, Total	EPA 6010D
	EPA 6020B
Copper, Total	EPA 6010D
	EPA 6020B
Iron, Total	EPA 6010D

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Metals I

Iron, Total	EPA 6020B
Lead, Total	EPA 6010D
	EPA 6020B
Magnesium, Total	EPA 6010D
	EPA 6020B
Manganese, Total	EPA 6010D
	EPA 6020B
Nickel, Total	EPA 6010D
	EPA 6020B
Potassium, Total	EPA 6010D
	EPA 6020B
Silver, Total	EPA 6010D
	EPA 6020B
Sodium, Total	EPA 6010D
	EPA 6020B
Strontium, Total	EPA 6010D
	EPA 6020B

Metals II

Aluminum, Total	EPA 6010D
	EPA 6020B
Antimony, Total	EPA 6010D
	EPA 6020B
Arsenic, Total	EPA 6010D
	EPA 6020B
Beryllium, Total	EPA 6010D
	EPA 6020B
Chromium VI	EPA 7196A

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Metals II

Mercury, Total	EPA 7471B
Selenium, Total	EPA 6010D
	EPA 6020B
Vanadium, Total	EPA 6010D
	EPA 6020B
Zinc, Total	EPA 6010D
	EPA 6020B

Metals III

Cobalt, Total	EPA 6010D
	EPA 6020B
Molybdenum, Total	EPA 6010D
	EPA 6020B
Thallium, Total	EPA 6010D
	EPA 6020B
Tin, Total	EPA 6010D
	EPA 6020B
Titanium, Total	EPA 6010D
	EPA 6020B

Minerals

Bromide	EPA 9056A
Chloride	EPA 9251
	EPA 9253
	EPA 9056A
Fluoride, Total	EPA 9214
	EPA 9056A
Sulfate (as SO4)	EPA 9038

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Minerals	
Sulfate (as SO4)	EPA 9056A
Miscellaneous	
Boron, Total	EPA 6010D
	EPA 6020B
Cyanide, Total	EPA 9012B
Organic Carbon, Total	Lloyd Kahn Method
Phenols	EPA 9065
Specific Conductance	EPA 9050A
Sulfide (as S)	EPA 9034
Nitroaromatics and Isophorone	
2,4-Dinitrotoluene	EPA 8270E
2,6-Dinitrotoluene	EPA 8270E
Isophorone	EPA 8270E
Nitrobenzene	EPA 8270E
Pyridine	EPA 8270E
Nitrosoamines	
N-Nitrosodimethylamine	EPA 8270E
N-Nitrosodi-n-propylamine	EPA 8270E
N-Nitrosodiphenylamine	EPA 8270E
Nutrients	
Nitrate (as N)	EPA 9056A
Nitrite (as N)	EPA 9056A
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015D
Gasoline Range Organics	EPA 8015D

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Phthalate Esters

Benzyl butyl phthalate	EPA 8270E
Bis(2-ethylhexyl) phthalate	EPA 8270E
Diethyl phthalate	EPA 8270E
Dimethyl phthalate	EPA 8270E
Di-n-butyl phthalate	EPA 8270E
Di-n-octyl phthalate	EPA 8270E

Polychlorinated Biphenyls

Aroclor 1016 (PCB-1016)	EPA 8082A
Aroclor 1016 (PCB-1016) in Oil	EPA 8082A
Aroclor 1221 (PCB-1221)	EPA 8082A
Aroclor 1221 (PCB-1221) in Oil	EPA 8082A
Aroclor 1232 (PCB-1232)	EPA 8082A
Aroclor 1232 (PCB-1232) in Oil	EPA 8082A
Aroclor 1242 (PCB-1242)	EPA 8082A
Aroclor 1242 (PCB-1242) in Oil	EPA 8082A
Aroclor 1248 (PCB-1248)	EPA 8082A
Aroclor 1248 (PCB-1248) in Oil	EPA 8082A
Aroclor 1254 (PCB-1254)	EPA 8082A
Aroclor 1254 (PCB-1254) in Oil	EPA 8082A
Aroclor 1260 (PCB-1260)	EPA 8082A
Aroclor 1260 (PCB-1260) in Oil	EPA 8082A
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1262 (PCB-1262) in Oil	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A
Aroclor 1268 (PCB-1268) in Oil	EPA 8082A

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Polynuclear Aromatic Hydrocarbons

Acenaphthene	EPA 8270E
Acenaphthylene	EPA 8270E
Anthracene	EPA 8270E
Benzo(a)anthracene	EPA 8270E
Benzo(a)pyrene	EPA 8270E
Benzo(b)fluoranthene	EPA 8270E
Benzo(g,h,i)perylene	EPA 8270E
Benzo(k)fluoranthene	EPA 8270E
Chrysene	EPA 8270E
Dibenzo(a,h)anthracene	EPA 8270E
Fluoranthene	EPA 8270E
Fluorene	EPA 8270E
Indeno(1,2,3-cd)pyrene	EPA 8270E
Naphthalene	EPA 8270E
Phenanthrene	EPA 8270E
Pyrene	EPA 8270E

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270E
2,4,5-Trichlorophenol	EPA 8270E
2,4,6-Trichlorophenol	EPA 8270E
2,4-Dichlorophenol	EPA 8270E
2,4-Dimethylphenol	EPA 8270E
2,4-Dinitrophenol	EPA 8270E
2-Chlorophenol	EPA 8270E
2-Methyl-4,6-dinitrophenol	EPA 8270E
2-Methylphenol	EPA 8270E
2-Nitrophenol	EPA 8270E

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Priority Pollutant Phenols

3-Methylphenol	EPA 8270E
4-Chloro-3-methylphenol	EPA 8270E
4-Methylphenol	EPA 8270E
4-Nitrophenol	EPA 8270E
Pentachlorophenol	EPA 8270E
Phenol	EPA 8270E

Semi-Volatile Organics

1,1'-Biphenyl	EPA 8270E
1,2-Dichlorobenzene, Semi-volatile	EPA 8270E
1,3-Dichlorobenzene, Semi-volatile	EPA 8270E
1,4-Dichlorobenzene, Semi-volatile	EPA 8270E
2-Methylnaphthalene	EPA 8270E
Acetophenone	EPA 8270E
Benzaldehyde	EPA 8270E
Benzoic Acid	EPA 8270E
Benzyl alcohol	EPA 8270E
Caprolactam	EPA 8270E
Dibenzofuran	EPA 8270E

Volatile Aromatics

1,2,4-Trichlorobenzene, Volatile	EPA 8260D
1,2,4-Trimethylbenzene	EPA 8260D
1,2-Dichlorobenzene	EPA 8260D
1,3,5-Trimethylbenzene	EPA 8260D
1,3-Dichlorobenzene	EPA 8260D
1,4-Dichlorobenzene	EPA 8260D
2-Chlorotoluene	EPA 8260D

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All approved analytes are listed below:

Volatile Aromatics

4-Chlorotoluene	EPA 8260D
Benzene	EPA 8260D
Bromobenzene	EPA 8260D
Chlorobenzene	EPA 8260D
Ethyl benzene	EPA 8260D
Isopropylbenzene	EPA 8260D
m/p-Xylenes	EPA 8260D
Naphthalene, Volatile	EPA 8260D
n-Butylbenzene	EPA 8260D
n-Propylbenzene	EPA 8260D
o-Xylene	EPA 8260D
p-Isopropyltoluene (P-Cymene)	EPA 8260D
sec-Butylbenzene	EPA 8260D
Styrene	EPA 8260D
tert-Butylbenzene	EPA 8260D
Toluene	EPA 8260D
Total Xylenes	EPA 8260D

Volatile Chlorinated Organics

Benzyl chloride	EPA 8260D

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 8260D
1,1,1-Trichloroethane	EPA 8260D
1,1,2,2-Tetrachloroethane	EPA 8260D
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260D
1,1,2-Trichloroethane	EPA 8260D
1,1-Dichloroethane	EPA 8260D

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Volatile Halocarbons

1,1-Dichloroethene	EPA 8260D
1,1-Dichloropropene	EPA 8260D
1,2,3-Trichloropropane	EPA 8260D
1,2-Dibromo-3-chloropropane	EPA 8260D
1,2-Dibromoethane	EPA 8260D
1,2-Dichloroethane	EPA 8260D
1,2-Dichloropropane	EPA 8260D
1,3-Dichloropropane	EPA 8260D
2,2-Dichloropropane	EPA 8260D
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260D
2-Chloroethylvinyl ether	EPA 8260D
3-Chloropropene (Allyl chloride)	EPA 8260D
Bromochloromethane	EPA 8260D
Bromodichloromethane	EPA 8260D
Bromoform	EPA 8260D
Bromomethane	EPA 8260D
Carbon tetrachloride	EPA 8260D
Chloroethane	EPA 8260D
Chloroform	EPA 8260D
Chloromethane	EPA 8260D
cis-1,2-Dichloroethene	EPA 8260D
cis-1,3-Dichloropropene	EPA 8260D
Dibromochloromethane	EPA 8260D
Dibromomethane	EPA 8260D
Dichlorodifluoromethane	EPA 8260D
Hexachlorobutadiene, Volatile	EPA 8260D
Methyl iodide	EPA 8260D

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Volatile Halocarbons

Methylene chloride	EPA 8260D
Tetrachloroethene	EPA 8260D
trans-1,2-Dichloroethene	EPA 8260D
trans-1,3-Dichloropropene	EPA 8260D
trans-1,4-Dichloro-2-butene	EPA 8260D
Trichloroethene	EPA 8260D
Trichlorofluoromethane	EPA 8260D
Vinyl chloride	EPA 8260D

Volatile Organics

1,4-Dioxane	EPA 8260D
	EPA 8270E
2-Butanone (Methylethyl ketone)	EPA 8260D
2-Hexanone	EPA 8260D
2-Nitropropane	EPA 8260D
4-Methyl-2-Pentanone	EPA 8260D
Acetone	EPA 8260D
Acetonitrile	EPA 8260D
Carbon Disulfide	EPA 8260D
Cyclohexane	EPA 8260D
Di-ethyl ether	EPA 8260D
Ethyl Acetate	EPA 8260D
Isobutyl alcohol	EPA 8260D
Isopropanol	EPA 8260D
Methyl acetate	EPA 8260D
Methyl cyclohexane	EPA 8260D
Methyl tert-butyl ether	EPA 8260D
n-Butanol	EPA 8260D

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Volatile Organics

o-Toluidine	EPA 8270E
Propionitrile	EPA 8260D
tert-butyl alcohol	EPA 8260D
Tetrahydrofuran	EPA 8260D
Vinyl acetate	EPA 8260D

Sample Preparation Methods

EPA 5035A-L
EPA 5035A-H
EPA 3580A
EPA 9030B
EPA 3050B
EPA 3550C
EPA 3546
EPA 3060A

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Title: The Analysis of Digestates for Hexavalent Chromium by EPA SW846 7196A

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Jasmine Parillo 06/16/20	Dan Helfrich 06/17/20
Jasmine Parillo Date	Dan Helfrich Date
Department Manager	Health & Safety Manager /Coordinator
Carl Armbruster 06/16/20	Mark Acierno 06/17/20
Carl Armbruster Date	Mark Acierno Date
Quality Assurance Manager	Laboratory Director

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This SOP is applicable to the determination of hexavalent chromium digestates of solid samples using SW846 Method 7196A. Solid samples must be first digested according to Method 3060A USEPA SW846 3rd Edition, TestAmerica Edison SOP ED-WET-010 (The Alkaline Digestion of Soil Samples for the analysis of hexavalent chromium).

1.1.2 The laboratory's reporting limit for hexavalent chromium in soil is 2.0 mg/kg.

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Solid samples are digested utilizing SOP ED-WET-010, alkaline digestion of soil samples via Method 3060A. Following the sample preparation and digestion procedure, dissolved hexavalent chromium is determined spectrophotometrically at 540 nm by reaction with diphenylcarbazide in acid solution.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Molybdenum and Mercury react to form color with diphenylcarbazide, however the intensity of color is much lower than those for chromium at the specified pH. Concentrations up to 200 mg/L can be accepted.

4.2 Vanadium interferes strongly, but can be tolerated up to 10 times the concentration of Cr (VI) present.

4.3 Iron may cause a yellow color but should not affect the colorimetric measurement at 540 nm.

4.4 Reducing matter may reduce hexavalent Cr to trivalent Cr in varying amounts. No preventative measure is available at this time; however, interference is checked by post digestion spike samples.

4.4.1 The reducing/oxidizing tendency of each matrix may be determined by characterization of samples for additional analytical parameters, such as pH, ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon

(TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

4.5 For waste materials or soils containing soluble Cr (Ill) concentrations greater than four times the laboratory Cr (VI) reporting limit, Cr (VI) results obtained using this method may be biased high due to method-induced oxidation.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at an elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

6.0 Equipment and Supplies

6.1. Instrument

- Spectrophotometer for use at 540 nm, providing a light path of 1cm or longer
- pH meter, Orion benchtop or equivalent

6.2. Supplies

- Nalgene 115-ml disposable filter unit with 0.45um filter
- Membrane filters, 0.45um and 0.1 um
- Specimen cups (plastic)
- Eppendorf or Finnpipettes, varying volumes
- Class A volumetric flasks
- Class A graduated cylinder
- Vacuum pump
- Magnetic stirrer (Teflon coated)
- Transfer pipettes

7. Reagents and Standards

7.1. Reagents

7.1.1. Sulfuric acid (10% v/v): Measure 100 ml of concentrated reagent grade H_2SO_4 into a 1000 ml volumetric flask (1/2 filled w/D.I. water) and dilute to mark with deionized water. Stable for six months, store at room temperature.

7.1.2. Indicator solution (DPC): Place 5.0 g 1,5-Diphenylcarbazide (AR Grade) in an amber 1000 ml volumetric flask and dilute to mark with acetone. Prepare monthly or when solution becomes cloudy. Store at room temperature.

7.1.3. Acetone: reagent grade; for stability and storage information refer to manufacturer's instructions.

7.1.4. 1:1 HNO3: Add 500ml DI to a 1 Liter volumetric flask and then slowly add 500ml of concentrated reagent grade HNO3. Solution is stable for 6 months. Store at room temperature.

7.1.5. 1N NaOH: Place 4.0g reagent grade NaOH in a 100ml volumetric flask and dilute to the mark with deionized water. Stable for six months, store at room temperature.

7.2 Standards

7.2.1 Potassium Dichromate Primary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3093-01 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. Stable for 6 months, store at room temperature.

7.2.2 Potassium Dichromate Secondary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3090-04 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. **Note**: Prepare the secondary standard from a source or lot different from the primary standard solution. Stable for 6 months, store at room temperature.

7.2.3 Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6): Prepare by adding 10 ml of 1000 mg/L Cr+6 primary standard (Sec 7.2.1) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.4 Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) Prepare by adding 10 ml of 1000 mg/L Cr+6 secondary standard (Sec 7.2.2) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.5 Lead Chromate: AR Grade; for stability and storage information refer to manufacturer's instructions.

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container [1]	Min. Sample Size[2]	Preservation	Holding Time [3]	Reference
Soils	Glass, plastic	2.5 grams	Cool 4 ± 2°C	30 Days - *from sampling to extraction*	SW846 Method 3060A
				168 Hours – *from extraction to determinative analysis*	SW846 Method 7196A

[1] Containers should not contain stainless steel
[2] Additional volume may be required if the sample is chosen for QA or if a re-prep is required.
[3]The alkaline digestate must be analyzed within 168 hours after extraction from the soil.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of 20 samples or less.

9.1.1. Method Blank: One method blank must be digested and analyzed per batch. A method blank consists of 50 ml digestion solution and is carried through the entire digestion and analytical procedure. The concentration of the method blank must be less than the reporting limit or the entire batch must be re-digested and re-analyzed.

9.1.2. Laboratory Control Sample: Two secondary source (LCS) shall be digested and analyzed per batch.

- **LCS soluble (LCSSRM)** is obtained from an independent source (Phenova). Prepare the LCS soluble solution by diluting the concentrated LCS solution as indicated in the manufacturer's instructions. If necessary, prepare a different dilution so that the concentration after analysis falls within the calibration curve. Add 5ml of the diluted LCS soluble solution into 50ml digestion solution. The results must be within vendor specified QC limits or the entire batch must be redigested and re-analyzed. Document the preparation of the LCS soluble solution in the Reagent module in TALS.

- **LCS insoluble (LCSI)** is prepared by adding 0.010g-0.020g of PbCrO4 into 50ml digestion solution. The results must be within 80-120% of the true value or the entire batch must be redigested and re-analyzed. (Typical amount of $PbCrO_4$ added to LCSI is 0.011g, this is equivalent to 708 mg Cr (VI)/Kg).

9.1.3. Predigestion Matrix Spikes: Both a soluble and insoluble pre-digestion matrix spikes must be analyzed per batch of ≤ 20 field samples.

9.1.3.1 Soluble matrix spike (MSS): spike the sample with 1.0 ml of the 100 ppm Cr (VI) primary spiking solution prepared in Sec 7.2.3 (equivalent to 40mg/kg Cr(VI)).

9.1.3.2 Insoluble matrix spike (MSI): to a separate sample aliquot add 0.010g- 0.020g of $PbCrO_4$. It is used to evaluate the dissolution during the digestion process. Typical amount of $PbCrO_4$ added to LCSI is 0.011g, this is equivalent to 708 mg Cr (VI)/Kg.

9.1.3.3 Both matrix spikes are then carried through the digestion process. The acceptance range for the matrix spike recoveries is 75-125%. If the matrix spike recoveries fall outside these recovery limits, the entire batch must be rehomogenized, redigested, and reanalyzed.

- Note: If redigesting/reanalyzing the batch, the Soluble matrix spike (MSS) is spiked at twice the sample concentration or 40mg/kg Cr(VI) whichever is greater.

9.1.3.4 If upon reanalysis, the matrix spike is not within the recovery limits of 75%-125%, but the LCS is within the criteria specified in Section 9.1.2, additional laboratory characterization of each sample in the batch for ORP (see TestAmerica Edison SOP ED-WET-066, Redox by SM2580) and pH (see TestAmerica Edison SOP ED-WET-061, pH Soil by SW846 9045C) may be required by the client to determine if the sample exhibits reducing conditions. Further characterization for total sulfides, Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), or Biochemical Oxygen Demand (BOD) may also be required by the client.

9.1.4. Duplicate (DU): One duplicate laboratory sample must be analyzed per batch. The sample used for the predigestion spike should be used for this purpose. Duplicate samples must have a Relative Percent Difference (RPD) of ≤20%, if both the original and the duplicate are ≥ four times the laboratory reporting limit. A control limit of ± 2.0mg/kg (laboratory reporting limit) is used when either the original or the duplicate sample is < four times the laboratory reporting limit.

9.1.5. Post Digestion Spike (PDS): Following the analysis (colorimetric determination), a post-digestion spike must be analyzed per batch. The spike concentration must be equivalent to 40mg/kg or twice the sample concentration, whichever is greater. To spike the extract with 40mg/Kg Cr

(VI), add 0.50 ml of 100 ppm Cr (VI) primary standard to a 50.0 ml alkaline extract, pour out 45.0 ml of the spiked sample and add color indicator and adjust pH (see Sec 10.3.4 & 10.3.5); bring the final volume to 50ml with deionized water. The post digestion spike must be performed on a field sample, not on a field blank or preparation blank. If possible perform the post digestion spike analysis on the sample used for the matrix spike. Recovery limits for the post-digestion spike are 85-115% of the true value. If the post digestion spike fails to meet the recovery limits, a post spike must be re-analyzed using the alkaline extract. Adjust the pH of the sample Duplicate (alkaline extract) back up to 8.0-8.5 using 1N NaOH; then respike and reanalyze following Sec. 10.3.4-10.3.5.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): The ICV (0.50 mg/L Cr (VI)) is analyzed immediately after an acceptable initial calibration. The ICV standard must be from a source separate from the calibration standards (i.e., different manufacturer or separate lot) and its recovery must be within ±10% of the expected value. The ICV is prepared and analyzed similar to the calibration standards; prepare by adding 0.5 ml of 100 ppm Cr (VI) Secondary Spiking Solution (Sec. 7.2.4) into a specimen cup containing 50 ml of digestion fluid. If the measured concentration exceeds the ±10% limit, a second analysis should be performed. If the result still exceeded the ±10% limit, the analysis should be terminated until the source of the problem is identified and corrected.

9.2.2. Continuing Calibration Verification (CCV): A CCV is a mid-point calibration Cr (VI) standard (0.5ppm) from a different source than the calibration standards. Add 0.5 ml of 100 ppm Cr (VI) Secondary Spiking Solution (Sec. 7.2.4) into a specimen cup containing 50 ml of digestion fluid. The CCV is analyzed after every 10 samples (20 readings including backgrounds), and after reading the last sample. Acceptance criteria for the CCV are 90-110% of the true value. If the result is not within the acceptance limits, all samples following the last acceptable CCV must be re-analyzed.

9.2.3. Initial Calibration Blank and Calibration Check Blank (ICB,CCB): An initial calibration blank (ICB) must be analyzed immediately following the calibration curve and a continuing calibration blank (CCB) is analyzed after each CCV. The sample consists of 50 ml digestion solution and is carried through the entire analytical procedure. The concentration of the CCB must be below the reporting limit if not, all samples following the last acceptable CCB must be re-analyzed.

10. Procedure

10.1. Sample Preparation:

10.1.1. Just prior to analysis, SLOWLY adjust the pH of the sample extract to a pH of 7.0-8.0 with constant stirring using 1:1 nitric acid. Record pH and time. If a flocculent precipitate forms after the addition of 1:1 HNO3, filter the sample through a 0.45 um membrane filter, a larger size filter may be used to pre-filter the sample. Monitor the pH with a calibrated pH meter. If the pH drops below 7.0, discard the sample extract and re-digest the sample.

10.1.1.1. Remove and rinse the stir bar. Quantitatively transfer the sample extract to a 100 ml volumetric flask and dilute to the mark with deionized water.

10.1.1.2. If necessary, the solution can be diluted to eliminate the effects of color on the analytical determination. Use the smallest dilution necessary and record results.

10.2. Calibration

10.2.1. Prepare fresh calibration standards everyday or before each analysis.

10.2.2. Prepare a set of 6 calibration standards. Prepare 0.0, 0.05, 0.1, 0.5, 0.75, and 1.25 mg/l $K_2Cr_2O_7$ standards by aliquoting 0, 0.05, 0.1, 0.5, 0.75 and 1.25 mls of 100ppm Cr (VI) Primary Spiking Solution (Sec 7.2.3) respectively into the specimen cups containing 50mls of digestion fluid. At the same time prepare the mid-point Initial Calibration Verification and Initial Calibration Blank, see Sec 9.2.

10.2.3. Adjust pH to 7.5±0.5 with 1:1 HNO_3 and dilute to 100 ml with DI water. Record pH reading and analysis time in the logbook.

10.2.4. Develop the color for the standards as explained in Section 10.3 of this SOP.

10.2.5. Following the preparation of the calibration standards, “zero” the spectrophotometer with the 0 ppm standard. Analyze the standards.

10.2.6. The correlation coefficient must be 0.995 or better.

10.2.7. Immediately following calibration of the spectrophotometer and before reading samples, verify the calibration of the spectrophotometer by analyzing an ICV and ICB.

10.3. Sample Analysis

10.3.1. Warm up spectrophotometer and set to 540nm.

10.3.2. Prepare two rows of plastic cups and label them accordingly (first row with sample no. and the 2nd row as background).

10.3.3. Pour out 45.0 ml of the sample extracts, which have already been pH adjusted to between 7.0 and 8.0, into the appropriately labeled cups. Do the same for the background cups using 10 ml of the sample extract.

10.3.4. Add 1.0 ml indicator solution and swirl. (Do not add reagent to background sample).

10.3.5. Slowly add 10% H_2SO_4 (Sec 7.1.1) dropwise while swirling and monitoring pH until pH is 2 ±0.5. Record reading in the appropriate logbook.

10.3.6. Transfer sample to 50 ml volumetric flask and bring to volume with deionized water. Empty contents into labeled specimen cup to mix.

10.3.7. Let stand 5-10 minutes for full color development.

10.3.8. Read calibration standards, QC samples, and samples in a 1 cm cell on a spectrophotometer at 540 nm. (Note: Calibration standards and samples must be read within the same timeframe following full color development).

10.3.9. Continue analyzing samples and quality control samples by first reading the sample followed by its background sample.

10.3.10. Dilute samples that exceed calibration curve using deioinized water and analyze following Sec. 10.3.3-10.3.7. Add dilution factor to TALS under "dilution" column. Note: The dilution should be made from the sample extract that has not had the indicator added.

10.3.11. After every twenty readings (10 samples) and after reading the last sample in the batch, verify the spectrophotometer calibration by reading a calibration check standard (CCV) and calibration check blank (CCB).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration = Hexavalent Chromium in mg/Kg = $\frac{A \times B \times E * 100}{C \times D}$

Where:

A = Concentration from the calibration curve in mg/L
B = Final digested volume in Liters
C = Wet sample weight in kg
D = percent solids
E = Dilution (if necessary)

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

11.4. Trivalent Chromium Calculation: When required, the calculation for trivalent chromium is completed using TALS Method '7196a_CR3.' Samples must be logged in under this method as well as the methods for Total chromium (metals) and Hexavalent chromium. Upon completion of the total and hexavalent chromium analyses the analyst may process the trivalent chromium results as follows:

- In order for TALS to perform an automated trivalent chromium calculation enter '1' (for 'yes') in trivalent chromium batch information page in response to the prompt 'automatically perform calculation.' As long as both methods are at 2nd level review status in TALS the program will automatically pull in the metals results for chromium and the hexavalent chromium results to perform the automated calculation. TALS will display an 'ok' message on the worksheet tab if the Cr6 result is less than Total Cr result.
- In cases where Total Cr results are not linked to TALS method 7196a_CR3, trivalent chromium is calculated manually. Enter '0' (for 'no') in batch information page 'Perform calculation.' Results will be entered manually.

11.5. Data reduction:

11.5.1. All data is recorded directly in TALS and recorded in the logbook at the time the analysis is performed.

11.5.2. Attach the prep and analytical logbook pages and calibration curve to the batch as a pdf file.

11.5.3. On the worksheet tab, enter the sample absorbance under 'Uncorrected Abs' and background absorbance under 'Color Blank Abs.' Also, enter the pH between 7-8 as 'initial pH',' pH between 1.5-2.5 as 'Final pH' and the background pH under 'Notes.'

11.5.4. Record special notes and observations in the "worksheet" tab and record reagent information in the prep batch information page (see "view batch information" page of ADII).

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.5.5.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3. Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.4. Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

11.5.6. Analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. After the batch is second level reviewed, the checklist is filed in wetchem department and scanned in the network drive.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are

disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.
- Sample waste-After analysis, the samples and standards are collected in a polyethylene container labeled 'Cr6+ waste.' Once the container is full, it is brought to the waste room for disposal.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 7196A.

15.2. Test America Edison SOP ED-WET-010, The Alkaline Digestion of Soil Samples for the Analysis of Hexavalent Chromium, most current revision.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. TestAmerica Edison SOP ED-WET-066, Redox, Analysis of Oxidation-Reduction Potential, most current revision.

15.7. TestAmerica Edison SOP ED-WET-061, Analysis of pH for soil and organic samples electrochemically, Method 9045C, most current revision.

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16. Method Modifications:

N/A

17. Attachments

N/A

18. Revision History

- **Revision 12, dated 17 June 2020**
  - Updated SOP header with Eurofins emblem.
  - Sec. 9.1.2: Changed LCS source vendor from ERA to Phenova.
  - Sec. 11.5.5: Revised data review procedure to include TALS DRC procedures.
  - Sec 15.8: Deleted Wetchem Data Review Checklist Work Instruction # EDS-WI-008, not applicable.

- **Revision 11, dated 05 March 2018**
  - Sec 11.4: Added procedure for Trivalent chromium calculation using TALS method 7196a_CR3; subsequent section adjusted accordingly.

- **Revision 10, dated 23 February 2016**
  - Sec 9.1.2 and 9.1.3.2: Revised the allowable amount of PbCrO4 to add to the QC sample LCSI to reflect the range referenced in the Method. Also included the typical amount of $PbCrO_4$ that is added to the QC sample (LCSI and MSI).
  - Sec 9.1.3.3: Added spiking instructions for MSS when redigesting a batch; MSS should be spiked at 2x sample concentration or 40mg/kg, whichever is greater.

- **Revision 9, dated 25 September 2013**
  - Sec 1 & 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec. 9.1.2: Changed nomenclature to LCSSRM and revised the acceptance limits from 85-115% to vendor specified QC limits.
  - Sec. 9.1.3.3: Deleted text: '*Note: If the spiked sample concentration is greater than 4X the predigestion spike concentration, no redigestion/reanalysis is required unless the samples are from a NJ site in which case the project manager should be contacted for further guidance.'*

    Per method 3060a, all batches must be rehomogenized, redigested and reanalyzed if MSS or MSI fail regardless of location.
  - Sec. 10.3.5: Changed pH range from 1.6-2.2 to pH 2.0 +/-0.5 as per the method.
  - Sec. 11.4.3: Changed pH range from 1.6-2.2 to pH 1.5-2.5 as per the method.

- **Revision 8, dated 06 September 2011**
  - Sec 3: Revised the LQM reference for the list of definitions.
  - Sec 9.1.3.4: Clarified lab procedures for failing matrix spikes.
  - Sec. 9.2.2: Added procedure if the CCV is not within acceptance limits.
  - Sec. 9.2.3: Added procedure if CCB is not below reporting limit.
  - Sec. 11.4: Revised data reduction section to reflect actual laboratory practices.
  - Sec 15.9: Reference added.

- **Revision 7, dated 25 August 2009**

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec 1.1: added laboratory's reporting limit; change the range of analysis to 0.05-1.25mg/L to reflect actual laboratory practices.
  - Sec 9.1.5: Added preparation instruction for Post digestion spike.
  - Sec. 9.2: added ICV in the Instrument QC.
  - Sec 10.1: Added procedure for adjusting pH of sample extract; procedure formerly described in the Digestion SOP (ED-WET-010).
  - Sec 15: Added applicable references.

- **Revision 6a, dated 21 April 2009**

  - Sec.4.3: Added membrane filters, 0.45um and 0.1um.
  - Sec 4.4: Delete Erlenmeyer flasks and beakers
  - Sec 4.8:Add: Eppendorf or Finnipettes; varying volume
  - Sec 5.1, 5.2 & 5.4: Add stability and storage information.
  - Sec 5.5: Added reagent used for the pH adjusted PS: 1N NaOH: Place 4.0g NaOH in a 100 volumetric flask and dilute to the mark with deionized water. Solution is stable for 6 months. Store at room temperature.
  - Sec. 6.1: Potassium Dichromate Primary Standard Solution (1000mg/L Cr+6): Weight 0.2829g of dried (105ºC) potassium dichromate and dissolve in deionized water in a 100ml flask. Dilute to the mark. Store at room temperature. Solution is stable for 6 months.
  - Sec 6.1: Deleted pre-made standard purchased from Inorganic Ventures. Cr6+ Primary Standard will be prepared in the lab.
  - Sec 6.2: Specified source of the 1000 ppm Cr(VI).
  - Sec 6.3 and 6.6: Deleted the 10mg/L Cr (VI) working standard for soil analysis.
  - Sec 6.5: Add storage information.
  - Sec 6.4: Add text: Source must be different from the primary standard.
  - Sec 9.2.2: Replace 100 ppm stock standard with 100 ppm Cr6+ Primary spiking solution.
  - Sec. 9.2.3: Add text: Record pH reading in the logbook.
  - Sec. 9.3.8: Add text: Make the dilution from the sample that has not had the indicator added.
  - Sec 9.3.6: Add text: Before reading samples, verify the calibration of the spectrophotometer by analyzing a CCV and CCB.
  - Sec. 10.7: Added procedure for reanalyzing Post Spike sample to conform to method requirements: *If the post digestion spike fails to meet the recovery limits, a post spike must be reanalyzed using the alkaline extract. Adjust the pH of the sample Duplicate (alkaline extract) back up to pH 8.0-8.5 using 1N NaOH; then*

respike and reanalyze.

- **Revision 6, dated March 2007**

  - Section 7.2: Section deleted (start color development for digested samples within one hour after adjusting pH between 7.5±0.5); section not applicable.
  - Section 9.3.7: The text in italics was inserted to clarify the time period for measuring sample absorbance.

    "Note: Calibration standards and samples must be read within the same timeframe following full color development."
  - Section 10.2: Deleted the requirement that 'CCS is taken through the entire digestion process' to reflect actual laboratory practices.
  - Section 10.4. Revised to specify the types of LCS used including their source and acceptance limits.

- **Revision 5, dated 30 October 2006**

  - Section 9.2.2: Revised the high point of the calibration curve from 1.0 ppm to 1.25 ppm. Standards preparation instructions revised accordingly.
  - Section 9.2.2: Revised to include instructions for preparation of a second source mid-point Calibration Check Standard.
  - Section 10: Inserted a new Section 10.1 explaining the QA batching system and inserted a new section 10.4 detailing the LCS requirements. All existing sections renumbered accordingly.
  - Section 10.6 (previously Section 10.5): The italicized text was added to the following sentence:

    "NOTE: If the spiked sample concentration is greater than 4x the predigestion spike concentration, no redigestion/reanalysis is required *unless the samples are from a NJ site in which case the lab project manager should be contacted for further guidance."*
  - Section 10.6 (previously Section 10.5): Added STL Edison SOP and method references for ORP and pH.
  - Section 10.7 (previously Section 10.6): Added the following text: "If the post digestion spike fails to meet the recovery limits, a new aliquot of the sample must be re-spiked and re-analyzed."
  - Section 10.8 (previously Section 10.7): Revised first sentence to read "One duplicate laboratory sample per batch *must* be analyzed." ('Must' previously read 'should').

Title: Mercury Analysis for Solid and Semisolid Waste Samples using the Leeman Mercury Analyzer (Cold Vapor Technique) by SW846 Method 7471B

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

SW846 Method 7471B is applicable to the determination of total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be digested prior to analysis. If this digestion procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix.

The typical detection limit using a 0.6 gram sample size is 0.017 mg/Kg Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

The sample is digested as described in this SOP and is analyzed using cold vapor atomic absorption. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance at 253.7-nm is measured as a function of mercury concentration.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Potassium permanganate is added to eliminate possible interferences from sulfide.

4.2 Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/Kg Copper.

4.3 Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 254 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent (25 mL).

4.4 Certain volatile organic materials that absorb at 253.7 nm may also cause interference. The analysis of the undigested sample should determine if this type of interference is present.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/M^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Permanganate	Oxidizer	5 Mg/M^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

6.1.1. Leeman Laboratories Inc. Hydra IIAA Automated Hg Analyzer

6.1.2. Computer and Monitor with Leeman Envoy software.

6.1.3. Top loader balance, 300gm capacity, and minimum sensitivity of ±1.0 mg

6.1.4. Hotblock digestor: Adjustable and capable of maintaining a temperature between 95 ±3°C.

6.2. Supplies

6.2.1 Pipettes and tips in various sizes

6.2.2 100 ml volumetric flasks

6.2.3 15 ml sample cups

6.2.4 Pump tubing:

- Sample, viton, blue tab
- Reductant, red tab
- Drain, blue tab

- Rinse, Black tab

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6.2.5 Drying Tube – Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.

6.2.6 Nitrogen or Argon supply - capable of producing 80 PSI.

7.0 Reagents and Standards

Storage requirements: store at room temperature

Life of Reagent: Concentrated acids: refer to manufacturer's instructions
Laboratory prepared reagents and diluted acids: one year

Record reagent preparations in the TALS Reagent Module.

7.1 Reagents

7.1.1 Nitric acid - Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions

7.1.2 Hydrochloric acid-Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.3 Potassium Permanganate (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.4 Sodium Chloride (analytical reagent grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.5 Hydroxylamine Hydrochloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.6 Stannous Chloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.7 Deionized water - 18 megohm minimum

7.1.8 10% Hydrochloric Acid- Cautiously add 200 ml of concentrated HCl to a container and bring to final volume of 2 liters with deionized water. Store at room temperature; stable for one year.

7.1.9 Stannous chloride solution - Add 50 g of $SnCl_2$ to 500 ml 10% HCl solution. Store at room temperature; stable for one year.

7.1.10 Sodium chloride/Hydroxylamine Hydrochloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water. Store at room temperature; stable for one year.

7.1.11 Potassium permanganate ($KMnO_4$) 5% solution w/v - Dissolve 100 g of $KMnO_4$ in deionized water and dilute to 2 liters using deionized water. Store at room temperature; stable for one year.

7.1.12 0.15% Nitric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 12mL of concentrated HNO_3 and bring the final volume up to 8 liters with deionized water.

7.2 Standards

Storage requirements: all standards are stored at room temperature

Shelf-life:	Stock standards – refer to manufacturer's instructions Intermediate standards – made fresh daily Working standards – made fresh daily (Note: expiration date must not go beyond the expiration date of the source stock).
Concentration:	see Attachment 1 for example certificates of analysis (COA) of Hg standards listed below. The COA lists the manufacturer's Lot number, certified concentration and shelf life.

Document standard preparation in TALS, see Sec 11.5.3.

7.2.1 Stock Mercury Calibration (10 ppm Hg) - Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.2 Secondary Stock Mercury Calibration Standard (10 mg/L Hg) - Purchase from Inorganic Ventures; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.3 Intermediate Calibration Standard (DCAL-Int), 100 ug/L Hg: Dilute 1 ml of Hg calibration stock standard solution (Sec 7.2.1) to 100 ml with 0.15% HNO_3.

7.2.4 Intermediate Initial Calibration Verification Standard (DQCS-Int), 100 ug/L Hg: Dilute 1 ml of Hg stock Calibration Verification standard, 10ppm Hg (Sec 7.2.2) solution to 100 ml with 0.15% HNO_3.

7.2.5 Calibration Standards: Use six 50 ml hotblock cups to prepare the standards. Spike the calibrations standards cups as follows:

Calibration Standard	DCAL-Int Spike Volume (mL)	0.15% HNO_3 (mL)	Final Conc (ug/L)
Std1 (Cal Blk)	0	5	0
Std 2	0.1	4.9	0.2
Std 3	0.5	4.5	1
Std 4	1	4	2
Std 5	2.5	2.5	5
Std 6	5	0	10

Digest the standards following Sample preparation in Section 10.1.

7.2.6 Initial Verification Standard (ICV) 5.0 ppb: Using a hotblock cup, add 2.5 mL of 0.15 HNO_3 and 2.5 ml of DQCS-INT, (Sec 7.2.4). Digest the standard following Section 10.1. After digestion, the ICV will contain 5.0 ppb of Hg.

7.2.7 Continuing Calibration Verification Standard (CCV) 5.0 ppb: Follow the preparation instructions for Std 5 (see Sec 7.2.5)

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Plastic Glass	5 grams	Cool 4 ± 2ºC	28 Days	SW846 Method 7471B

[1] Inclusive of digestion and analysis.

Samples are to be analyzed without drying. A separate procedure is used to determine the percent solids in the sample.

For sample homogenization procedures refer to TestAmerica Edison SOP ED-GEN-007 (Subsampling).

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< RL; 5% of the regulatory limit; 5% of the measured concentration in the sample
Laboratory Control Soil Sample Reference Material (LCSSRM) in soil samples	1 in 20 or fewer samples	Vendor's certified limit
Matrix Duplicate (DUP)[1]	1 in 20 or fewer samples	If original sample and dup are both ≥ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Matrix Spike (MS)[1]	1 in 20 or fewer samples	80-120%
Matrix Spike Duplicate (MSD)[2]	1 in 20 or fewer samples	80-120%; If original sample and dup are both ≥ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Serial Dilution (SD)	1 in 20 or fewer samples	±10%

[1] The sample for DUP and MS are randomly selected, unless specifically requested by a client; Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.
[2] A MSD is prepared and analyzed when requested by the client.

9.1.1. Method Blank: One laboratory method blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). The method blank is used to identify possible contamination during acid digestion. Results must be less than the RL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample. If the analyte concentration in the method blank is above this control limit, the batch must be prepared again and the samples reanalyzed.

9.1.2. Laboratory Control Sample Soil Reference Material (LCSSRM: A laboratory control sample must be analyzed with each group of samples digested. For solid matrices, a vendor supplied solid matrix with certified values is carried through the same preparation procedure as the samples. The results of the solid LCS must fall within the 'QC Performance acceptance limits' of the reference material used for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.

9.1.3 Matrix Duplicate (DUP): A duplicate is analyzed for each batch of samples digested. If original sample and duplicate are both ≥ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL.

9.1.4 Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.5 mL of DCAL-Int (Sec 7.2.3). This is equivalent to 1.0 ppb Hg (on instrument). A recovery of 80-120% is required.

9.1.5 Matrix Spike Duplicate (MSD): A matrix spike duplicate is prepared and analyzed when requested by the client. A portion of sample is spiked with 0.5 mL of DCAL-Int (Sec 7.2.3). This is equivalent to 1.0 ppb Hg (on instrument). A recovery of 80-120% is required. If matrix spike sample and matrix spike duplicate sample are both ≥ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL

9.1.6 Serial Dilution (SD): A five fold serial dilution must be performed on one sample per batch. The sample should contain a sufficiently high concentration; minimally a factor of 25 times the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. The results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

9.2. Instrument QC

9.2.1 Initial Calibration Verification (ICV): Initial calibration is verified after calibration using an independent check standard at a concentration near

the mid-point of the calibration (5.0ppb); see Sec 7.2.6 for preparation instructions. The results must be within 10% of the true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.2 **Continuing Calibration Verification (CCV):** Calibration verification is performed after the calibration, after every 10 samples, and at the end of the run. Use a concentration of mercury at the midpoint of the calibration range (5.0 ppb). See Sec. 7.2.7 for preparation instructions. The value obtained must be within 10% of the true value. If not, stop the analysis and recalibrate. Re-analyze the previous ten samples following the last good calibration verification. .

9.2.3 **Initial and Continuing Calibration Blank (ICB/CCB):** ICB/ CCB must be analyzed after the calibration curve, every 10 samples, and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

10 Procedure

10.1. Sample Preparation (includes all samples, standards, and blanks)

10.1.1 Mix the sample well and weigh 0.5 - 0.6 grams of sample (including the LCSSRM) and place in the bottom of an appropriately identified 50 mL hotblock cup. For QA samples, weigh three portions of 0.5 - 0.6 grams of sample and place in the bottoms of three hotblock cups labeled as SAMPLE, DUP, and MS. Before adding any reagents, spike the MS sample with 0.5 mL of DCAL-Int standard.

10.1.2 Except the calibration standards, add 5 mL of deionized water to all hotblock cups (i.e., all field samples, MB, LCSSRM, Dup, and MS).

10.1.3 Add 1.5 ml concentrated HNO_3 and 4.5 ml concentrated HCl. Heat 2 min on the hotblock at 95 $\pm$3°C. Cool.

10.1.4 Add 15 mL deionized water and 15 mL potassium permanganate solution (Sec 7.1.11) to each hotblock cup. Mix well. The same amount of $KMnO_4$ must be added to the standards and samples.

10.1.5 Cap the hotblock cups loosely enough so that pressure does not build up but also tight enough so that the caps stay on and that volume loss due to heating is minimized. Heat 30 min on the hotblock at 95 $\pm$3°C. Cool.

10.1.6 To each hotblock cup, add 6 ml Sodium chloride - Hydroxylamine hydrochloride solution to reduce excess permanganate.

10.1.7 Using deionized water, bring to a 50 mL final volume. Cap and mix well.

10.2. Calibration

10.2.1 The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in the in TALS reagent module.

10.2.2 Prepare the calibration standards and Calibration Verification Standards as stated in Sections 7.2.5, 7.2.6., & 7.2.7.

10.2.3. Calibration Curve Read-Back:

10.2.3.1. Low-Level Readback (at the RL) – evaluate the 0.2 ug/L calibration standard. The %RE (relative error) must be +/- 20% of the true value (see Sec 11.4). If %RE is outside of the criteria limits, stop the analysis and recalibrate.

10.2.3.2. Mid- Level Readback – evaluate the 5.0 ug/L calibration standard (the mid-level calibration standard). The %RE (relative error) must be +/- 10% of the true value (see Sec 11.4). If %RE is outside of the criteria limits, stop the analysis and recalibrate

10.3. Sample Analysis

10.3.1 Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.

10.3.2 The following analytical run sequence is currently used for samples analyzed under Method 7471B:

Instrument Calibration (Blank and five standards)
ICV
ICB
CCV
CCB

10 Samples
CCV
CCB
10 Samples
CCV
CCB
Repeat until run is complete
CCV
CCB

10.3.3 Instrument Operation:

10.3.3.1 Turn on instrument, computer and monitor.

10.3.4 Plumbing the Reagent Lines:

10.3.4.1 One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.

10.3.4.2 Reductant (Red); Connect tab end of tube to the reductant bottle and the other end to the bottom of the mixing tee.

10.3.4.3 Sample (Blue); Connect tab end of tube to the autosampler probe and the other end to the top of the mixing tee.

10.3.4.4 Drain (Blue) Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.

10.3.4.5 Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.

10.3.5 Preparation of Reagents:

10.3.5.1 Pour the $SnCl_2$ solution into the reductant bottle and connect to the red reductant tube connector.

10.3.5.2 Pour the ten percent HCl solution into the Rinse reservoir bottle.

10.3.6 Starting Program:

10.3.6.1 Click the Envoy icon on the computer desktop

10.3.6.2 Click Method, select 7471_7471B, and select OK
10.3.6.3 On the main screen, click the StartUp icon. Wait 15 minutes before analyzing calibrating.
10.3.6.4 Click Sequence Tab on bottom of screen
10.3.6.5 Click Sequence on top of screen
10.3.6.6 Select Open, then select New
10.3.6.7 Using the Prep Batch Sheet and hand scanner, enter the sample barcodes into the Sample ID column. Include the Serial Dilution and all needed sample dilutions
10.3.6.8 Pour out the digested calibration standards and samples into the proper locations on the autosampler.
10.3.6.9 Click the Run Sequence icon to begin calibration and to run samples.

10.3.7 Creating the Raw Data PDF:

10.3.7.1 Click the Analysis Tab, select Detailed

10.3.7.2 Click Load, select Use This To Make PDF

10.3.7.3 Click Report, Click Printer

10.3.7.4 Include the calibration curve graph:

10.3.7.4.1 Go to Methods Tab and click Calibration, click Print

10.3.7.5 Combine both documents using the PDF Creator program. The new document is located in the Documents folder on the C:\ drive. Add this document to the "Doc's" location in the analytical batch.

10.3.8 Shutting Down the Instrument:

10.3.8.1 Click the Stop icon on the main screen

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

11.3. Concentration: (mg/Kg) = $\frac{C \times V1 \times D}{W}$

Where: C= Element concentration from instrument (ppb)
V1= Final volume of sample digested (in liters)
D= Dilution performed on sample
W= Initial weight of sample digested (in gram)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4. Relative Error (%RE):

% Relative Error = (100) (MC-TC)/TC

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Processing:

11.5.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.5.2. Import Data to TALS

11.5.2.1. Click the Analysis tab
11.5.2.2. Select the analytical run that needs to be imported
11.5.2.3. Select Statistics
11.5.2.4. Click Load and select TALS Import, click OK
11.5.2.5. Click Report, click CSV File
11.5.2.6. Name the import file (e.g., batch name, today's date)
11.5.2.7. The newly created import file is in the Import Folder on the desktop. Send to TALS Import Folder

11.5.3. Sample and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation and expiration dates.

11.5.4. All reagents must be recorded in TALS Reagent Module.

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable preparation batch.

11.5.5.1. Open the preparation batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3. Acknowledge by filling in responses to all unacknowledged findings.

11.5.5.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.3.2. Fill in appropriate comments in the response box, then hit 'OK.'

11.5.5.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

11.5.6. Record the following reagents and the volume used for sample preparation in the batch information page under "batch comments" in TALS Analyst Desktop II: concentrated sulfuric acid, concentrated nitric acid, potassium permanganate, potassium persulfate and sodium chloride-hydroxylamine hydrochloride. Record reagents in TALS by opening the prep batch, click on "edit" and then right click to choose "view batch information." Enter the information in the "batch comments" section.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Instrument Detection Limit

The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. The IDL is determined by multiplying the average of the standard deviations obtained from the analysis of seven reagent blanks by 3.14.

12.3 Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.4 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

12.5. Lower Limit of Quantitation (LLOQ)

12.5.1. LLOQ verification: prior to analyzing samples under method 7470A, after any change that may affect the LLOQ, and quarterly, the LLOQ must be verified. The LLOQ is initially verified by the analysis of 7 replicate samples spiked at the LLOQ (0.2 ppb). The seven replicates must be digested prior to analysis. The LLOQ is verified when the mean recovery of the 7 replicates is +/-30% of the true value and the RSD < 20%.

12.5.2. LLOQ on-going verification: the LLOQ is re-verified quarterly by the analysis of a method blank (MB) and 1 blank sample (deionized water spiked at the LLOQ (0.2 ppb). The MB and LLOQ samples must be digested prior to analysis. The LLOQ is re-verified if the MB result is +/- 0.5 times the reporting limit for each analyte and when the recovery of the LLOQ sample is +/-30% of the .true value.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention.

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed.,U.S. Environmental Protection Agency, SW846 Manual, Method 7471B, Revision 2, January 1998.

15.2. Leeman Hydra IIAA Operating Manual.

15.3. TestAmerica Edison Document ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.

15.6. TestAmerica Edison Subsampling SOP, *ED-GEN-007,* most current revision.

15.7. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

16.0. Method Modifications:

Item	Method #	Modification
1 (Sec 10.3.5.1)	7471B	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's

		improvement that will reduce error due to loss of Mercury.
Sample preparation	7471B	The amount of DI water added to the sample at digestion is decreased to 15 ml from 50 ml (water bath procedure), while keeping the volume of reagents added during sample digestion the same. Sample final volume for hotblock procedure is modified to 50 ml (100 ml – water bath procedure) which allows for lesser sample volume hence less sample waste produced; hotblock digestion cup has maximum volume of 50 ml.

17.0. Attachments

Attachment 1: Certificate of Analysis of stock standards

18.0. Revision History

- Revision 5, dated 11 August 2022
  - Sec 12.5: Added the lower limit of quantitation (LLOQ), associated instructions and recovery limits.
- Revision 4, dated 03 August 2020
  - Updated SOP header with Eurofin logo.
  - Sec 6.1.1 & 15.2: Replaced Hydra AA with Hydra IIAA.
  - Sec 9.1 Table and 9.1.1: Revised MB control limits to <RL.
  - Sec 9.1, 9.1.4 and 9.1.5: Revised MS and MSD acceptance limits to 80-120%.
  - Sec 9.2.2: Revised CCV acceptance limits to +/-10%.
  - Sec 10.2.3: Added calibration curve read-back criteria.
  - Sec 11.4: Added Relative % error calculation.
  - Sec 11.5: Added Relative % error calculation.
  - Sec 11.5.6.: Added requirements to record reagent volumes in the batch information page of TALS ADII; subsequent sections adjusted accordingly.
- Revision 3, dated 12 October 2018
  - Sec 6.1.2: Removed WinHg instrument software reference and replaced with current instrument software, Envoy.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS in soil matrix
  - Sec 10.3.6: Updated the Starting Program instructions to reflect the new instrument software, Envoy.
  - Sec 10.3.7 & 10.3.8: Updated instructions for creating raw data pdf and shutting down instrument.
  - Sec 11.4.2: Updated instructions for importing data to TALS.
  - Sec 11.4.5: Replaced Metals Data Review Checklist with Data Review Checker (DRC) in TALS.

- Sec 15.8 & 15.9: Removed reference to EDS-WI-007 & EDS-WI-125; WI not applicable.

- Revision 2, dated 29 Nov 2016
  - Sec 6.1.4, Sec 10.1.3, & 10.1.5: updated the hotblock temperature range per method 7471B requirements
  - Sec 6.2.7: added Argon gas
  - Sec 7.2.4: the name of the intermediate ICV standard has been changed from DICV-Int to DQCS-Int
  - Sec 7.2.6: removed the CCV preparation instructions using the second source standard. The name of the intermediate ICV standard has been changed from DICV-Int to DQCS-Int
  - Sec 7.2.7: added preparation instructions for the CCV using the primary source standard
  - Sec 9.1: added the quality control sample MSD to the table
  - Sec 9.1.5: added quality control information for the MSD
  - Sec 11.4.5: Metals Data Review Checklist control # has been updated from ED-WI-007 to CA-Q-WI-042

- Revision 1, dated 12 May 2014
  - Sec 1.1: Revised the typical detection limit from 0.033 mg/kg to 0.017 mg/kg to reflect the 50mL final volume – hotblock procedure.
  - Sec 1 & 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 6.1.4: Added hotblock digestor to the list of equipment; removed autoclave from the list.
  - Sec 6.2: Added 50 mL hotblock digestion cups to replace the BOD bottles. Removed supplies which are no longer applicable (BOD bottles, graduated cylinders).
  - Sec 7.0 & 11.4.4: Removed Hg Reagent Logbook and replaced with TALS Reagent Module
  - Sec 7.1. Deleted Sulfuric Acid (previously referenced in Sec 7.1.1). Added 0.15% HNO_3 to list of reagents.
  - Sec 7.2.5: Updated Calibration standards' spiking instructions to reflect the 50 ml final volume.
  - Sec 7.2.6: Updated ICV/CCV spiking instructions to reflect the 50 ml final volume.
  - Sec 8.1: Added plastic container to the list of acceptable sample containers.
  - Sec 9.1 & 9.1.3: Clarified the acceptance limits for Matrix duplicate to reflect actual laboratory practices.
  - Sec 9.1.4: Revised the spiking amount added to the MS sample.
  - Replaced LCSS reference with LCSSRM in various sections of the SOP to reflect TALS QC type reference.
  - Sec 10.1: Revised sample preparation procedure to include hotblock digestor procedure; deleted all reference to autoclave digestion procedure.

- Sec 11.4.5 & 15.9: Added Work instructions EDS-WI-125 (TestAmerica Edison Metals Initial Calibration Data Review for Mercury).
- Sec 16: Added Sample prep method modification (i.e. final volume).

- Revision 0, dated 04 February 2011
  New

Attachment 1

Certificate of Analysis

Catalogue Number :	141-110-111/141-110-112/141-110-115
Description :	*Plasma*CAL - **ICP-MS Verification Standard 1 Solution B**
Lot Number :	**SC8298812**
Expiration Date :	**January 2010**

Analysis of Solution Standard by Inductively Coupled Plasma Spectroscopy (ICP-AES) traceable to NIST Standard Reference Materials : 3133

Actual Concentrations

Hg : 9.98 µg/ml

Matrix : **10% HNO_3**
Density : **1.040 g/ml @ 22.0°C**

MS 0882
Rec'd
1/14/09

Certified by : _____
Thomas Znoj, Chemistry Manager

Certification Date : **October 28, 2008**

This ICP-AES & ICP-MS Standard is guaranteed to be stable and accurate to within plus or minus 1.0% of the actual concentration up to the expiry date, provided the solution is kept tightly capped and stored under normal laboratory conditions. For these solutions, 18 megohm/cm double deionized water, high-purity acids, Class A glassware and acid-cleaned bottles are used. The Material Safety Data Sheet and this Certificate of Analysis are available on our web site. (Également disponible en Français)

Manufactured according to an ISO 9001:2000 Quality System and ISO 17025 (in-process)

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CERTIFICATE OF ANALYSIS

MS0879
Rec'd 1-12-09

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001:2000 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

2.0 **DESCRIPTION OF CRM** **10 µg/mL Mercury in 10% (v/v) HCL**

Catalog Number:	MSHG-10PPM
Lot Number:	**B2-HG02061**
Starting Material:	Hg metal
Starting Material Purity (%):	99.999549
Starting Material Lot No:	05214TX
Matrix:	10% (v/v) HCL

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 10.027 ± 0.020 µg/mL

Certified Density: 1.019 g/mL (measured at 22° C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
- This product is Traceable to NIST via an unbroken chain of comparisons to the following NIST SRMs:

Title: Closed System Purge and Trap and Extraction for Volatile Organics in Soil , SW846 Method 5035A

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Reddy Pakanati	04/11/19 Date	Dan Helfrich	04/11/19 Date
VOA Department Manager		Health & Safety Manager / Coordinator	
Carl Armbruster	04/11/19 Date	Mark Acierno	04/11/19 Date
Quality Assurance Manager		Laboratory Director	
Sylvanus Klusey	04/11/19 Date		
Operations Manager - Organics			

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method describes a closed system purge and trap procedure for the analysis of volatile organic compounds in soils, sediments and solid wastes.

1.1.2 This method is designed for use on samples containing low levels of volatile compounds (0.5 to 200ug/kg), and requires the use of hermetically sealed sample vials, which limits a samples exposure to the atmosphere, thereby minimizing the loss of volatile compounds.

1.1.3 Method 5035A can be used for most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Water-soluble compounds can be included in this technique, but will have higher quantitation limits due to poor purging efficiency.

1.1.4 Method 5035A also includes sample collection and preparation procedures for medium and high concentration soil samples (i.e., >200 ug/kg). Medium and high level soil samples are prepared either by field preservation of a soil sample in methanol or by creating a methanol extract from a soil sample that was collected as bulk soil.

1.1.5 The preparation steps described for medium and high level samples will be followed by the aqueous purge and trap procedure detailed in TestAmerica Edison SOP No. ED-MSV-001(*Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*).

1.1.6 The preparation and sample introduction steps described for low-level soils will be followed by analysis using TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) or ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*) as applicable.

1.1.7 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 **Low Level Soils:** Volatile organic compounds are determined by collecting a 5g-soil sample with a specially designed sampling device (ex., En Core® Sampler) in which the sample is stored without headspace. Upon receipt and within 48 hours of sampling, the soil is placed in a pre-weighed vial containing 5 mL of reagent water and a stir bar. The vial containing the soil is then frozen extending the hold time to 14 days. The same samples may be received from the field in a 40 mL vial with 5 mL water (ex., Terra Core® Sampler). Immediately prior to analysis, the soil is thawed, reagent water, surrogate spiking solution and/or internal standard

spiking solution are added to the vial by the automatic sampler, without opening the vial to the atmosphere. The sample vial is heated and purged with helium while being magnetically stirred. The volatile components travel through a transfer line to a sorbent trap. When purging is complete, the trap is heated and backflushed with helium to desorb the sample components on to a GC column for separation and analysis by the appropriate determinative method.

2.2 **Medium Level Soils:** Medium level soils are soils that were originally intended to be analyzed by a low level soil method, but show contaminants >200ug/kg when screened. A methanol extract is prepared by extracting 5g of soil with 10mL of methanol. The volatiles are effectively transferred from the soil to the methanol. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

2.2.1 Samples may also arrive from the field as 5 mL or 10 mL methanol preserved samples. These are considered to be medium level prepared samples.

2.3 **High Level Soils:** The high level soil method acts as a combined preservation and extraction technique. 25mL of methanol /surrogate solution is placed in each VOA vial and weighed prior to field sampling. Approximately 10g of soil is added to each vial in the field. Upon returning to the lab, the vial is weighed again, and the difference between the two weights is recorded as the initial weight of sample. As with the medium level method, the volatiles are effectively transferred to the methanol. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

2.4 **Waste Dilution:** Organic waste samples are prepared by extracting 1g of sample with 10mL of methanol / surrogate solution. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method and the determinative methods that follow are susceptible to contamination from a number of sources. Potential sources of contamination include organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatile laboratory is located in a separate building, away from the organic extraction area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatile laboratory.

4.3 The nitrogen used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 A trip blank prepared from organic-free reagent water is carried through the sampling, storage, and analysis of each group of samples to check for such contamination.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Purge vessels are removed from the autosampler units after each use, rinsed, baked, returned to the units and pre-purged before the next use.

4.7 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.8 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

4.9 Potential cross-contamination of samples stored in lab refrigerators is monitored by preparation, analysis and evaluation of storage blanks. Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After one week, the storage blanks are removed and analyzed. Additional details can be found in TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

5.1.1 Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required. A purge and trap unit consists of three parts: the sample purge unit, the trap, and the concentrator. Unit configurations currently in use are:

- OI Analytical 4551 Automatic Sampler/4560 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4560 concentrator;
- EST Centurion Autosampler/ EST Encon concentrator;
- Archon Autosampler/EST Encon concentrator.

6.1.2 A VOCARB 3000 trap from Supelco is used in the Encon concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001

6.1.3 An OI analytical purge trap #10 is used for the OI 4560 concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed to contain the following absorbents: Tenax/silica gel/carbon molecular sieve.

6.1.4 Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 Both the Encon and OI concentrators are capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.6 Gas chromatograph: HP 5890/Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: reference the applicable determinative method for column specifics.

6.1.8 Detector: Flame Ionization Detector or Mass Spectrometer (HP5971/5972/Agilent 5973). Reference the applicable determinative method for detector specifics.

6.1.9 Data system: HP Chemstation II for data acquisition and HP UNIX based TARGET software for data processing.

6.2 Equipment and Supplies

6.2.1 Freezer: Capable of holding a temperature of -7℃ to -20℃.

6.2.2 Refrigerator: Capable of holding a temperature of 4℃ ±2℃

6.2.3 Top loading analytical balance.

6.2.4 Portable field balance. Capable of weighing to 0.01g.

6.2.5 Microsyringes: 10 uL to 1000 uL.

6.2.6 Syringes: 5mL, 10mL, and 30mL gas-tight.

6.2.7 Volumetric flasks: Class “A” glassware, 10mL , 50mL , and 100 mL.

6.2.8 VOA vials: 40 mL glass with PTFE –faced septum.

6.2.9 En Core® Sampler. Designed to take a 5g-soil sample. Sealed to prevent loss of volatiles.

6.2.10 Vials: 2mL amber glass with screw cap with Teflon-faced septa.

6.2.11 Spatula: Narrow, stainless steel.

6.2.12 Stir bars: PTFE coated, small enough to spin freely inside a VOA vial.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Organic free reagent water. Distilled water purchased from Poland Spring.

7.1.2 Methanol. Purge and trap grade, purchased from JT Baker. (Cat # 9077-02)

7.1.2.1 Each lot of methanol and hydrochloric acid is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2 Standards

7.2.1 Calibration standard preparation and use is described in detail in each of the determinative methods.

7.2.2 Method 8260 Methanol/Surrogate solution (2.5 ppm) is prepared using purge and trap grade methanol and neat a,a,a-Triflurotoluene solution. Initially a primary solution is prepared at 2500 ppm:

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume of MeOH
1°Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4	Sigma Aldrich	 B67201 151998 396540	 1585 ul 2678 ul 1932 ul	Neat	2500ppm	1000 ml

The 2500 ppm solution is used to prepare a 2.5 ppm Method 8260 Methanol/Surrogate working solution by diluting 1.0 ml of 2500 ppm stock to 1000ml with purge and trap grade methanol.

7.2.3 Method 8015 (GRO) Methanol/Surrogate solution (1.25 ppm) is prepared by diluting 0.5ml of the 2500 ppm stock (see Section 7.1.4) to 1000 ml with purge and trap grade methanol.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Low Level Soils

Sample containers used to collect low-level soils are 40 mL VOA vials with PTFE-faced septa (closed system purge and trap), En Core® Samplers, Terra Core® kits or bulk soil.

8.1.1 For the 40 mL VOA vial (closed system purge and trap) collection procedure, add 1 clean magnetic stir bar and 5 mL of reagent water to each vial.

8.1.1.1. Cap the vial and weigh to the nearest 0.01g. Record this as the tare weight.

8.1.1.2. Collect approximately 5g of soil using an appropriate sampling device and add 5g to each of 3 vials for low level analysis.

8.1.1.3. Collect a fourth aliquot for field methanol preservation into a pre-weighed vial containing 10 mL of methanol.

8.1.1.4. Closed system purge and trap collection vials are re-weighed to determine actual sample weight. Record the sample weight on the sample vial.

8.1.1.5. Within 48 hours of sampling the collection vials are placed into a freezer (on their side to prevent breakage) at -7°C to -20°C. Freezing in this manner extends the holding time to 14 days from sampling.

8.1.2 For En Core® Sampler collection, collect up to 3 En Core® Sampler for low level preservation via freezing and a fourth for medium level preservation with methanol (Section 8.2 details the medium level preservation procedures for En Core® samplers). The low level preservation procedures are:

8.1.2.1. The En Core® device is received at the lab sealed without headspace with a cap and viton o-rings.

8.1.2.2. For each En Core® requiring low level preservation, add 5ml of reagent water and a magnetic stir bar to a 40 ml vial. Cap the vial.

8.1.2.3. Weigh each vial to the nearest 0.01g. Record this as the tare weight.

8.1.2.4. Transfer the contents of each En Core® to separate preweighed vials within 48 hours of sampling.

8.1.2.5. Reweigh the vial. Record the weight on each sample vial.

8.1.2.6. The vials are placed into a freezer (on their side to prevent breakage) at -7°C to -20°C. Freezing in this manner extends the holding time to 14 days from sampling.

8.1.2.7. A bulk soil sample must also be collected for the purposes of percent moisture analysis for dry weight determination.

8.1.2.8. Vials are removed from the freezer the day of analysis for thawing.

8.1.2.9. Stir bars may be reused after the vial is analyzed. Retrieve the stir bar from each vial using the magnetic stir bar retriever, rinse the stir bar with methanol and bake at 105°C for 2 hours.

8.1.3 Terra Core® Kits. This option requires samples to be field preserved:

8.1.3.1. The sample kit typically includes two pre-weighed 40 mL vials containing 5 mL of water with a small magnet stir bar and one or two pre-weighed 40 mL vials containing pre-measured and recorded amount of methanol.

8.1.3.2. Approximately 5g of sample is placed into each vial by the field sampler.

8.1.3.3. The vials containing the samples are re-weighed in the lab and the net sample weight is recorded for each vial. The vials containing the sample and water are frozen up until the time of analysis.

8.1.3.4. The methanol preserved samples are considered to be medium level prepared samples and do not require freezing but they must be refrigerated at 4℃ until time of analysis.

8.1.3.5. A bulk soil sample must also be collected for the purposes of percent moisture analysis for dry weight determination.

8.1.3.6. Stir bars may be reused after the vial is analyzed. Retrieve the stir bar from each vial using the magnetic stir bar retriever, rinse the stir bar with methanol and bake at 105°C for 2 hours.

8.1.4 Bulk soil. Typically consists of soil collected in 8-oz or 16-oz glass jars. At client request bulk soil samples can be sub-sampled, preserved and analyzed for volatile organics. Soils collected for low level VOA analysis as bulk soil in jars will be preserved as described above in Section 8.1.1. Documentation of preservation is recorded in the Soil Preservation Logbook.

8.2 Medium Level Soils

8.2.1 Medium level refers to any soil sample extracted in methanol not preserved in the field. The fourth Encore sample collected (as described in Section 8.1.2 above) will be prepared as a medium level soil in case the analysis of a low level preserved aliquot exhibits concentrations of target compounds exceeding 200 ug/kg. All medium level extracts must be refrigerated at 4℃ until time of analysis.

8.2.1.1. Prepare the medium level soil by adding 5 g of soil or Encore to 10 mL of Methanol / Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5. depending upon the method to be analyzed). All medium level extracts shall be prepared within 48 hours of collection. The weight of soil used to prepare the extract is recorded on the sample vial.

8.2.1.2. Since methanol extract results tend to increase with time as the sample contact time increases, a minimum contact time of one day is allowed ***or*** the soil is sonicated for 20 minutes.

8.2.1.3. Any soil requiring medium level preservation but collected out of compliance with method 5035A (bulk soil without preservative or not in an Encore device) will be prepared in the same manner as in section 8.2.1.1. Documentation will be noted in the soil preservation log.

8.3 High Level Soils

8.3.1 High level soils are field collected using laboratory prepared 40 ml VOA vials (with PTFE-faced septa) containing 25 mL of the Methanol/Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5. depending upon the method to be analyzed).

8.3.2 The vials containing the Methanol/Surrogate solution are weighed using a barcode reader and an analytical balance. The bottle ID and weight (Weight 1) are recorded in the methanol-tracking program.

8.3.3 The containers are issued to the field as required.

8.3.4 Field samplers are directed to weigh 10 g of sample using a field balance. They must carefully add the 10 g sample to the sample vial containing the Methanol/Surrogate solution, taking care not to spill any of the solution from the vial.

8.3.5 Field samplers are directed to wipe the neck of the vial carefully to remove any soil around the threads. This helps to ensure a good seal once the vial is capped. Field samplers are further directed to cap the vial, shake gently, dispersing the soil into the methanol. An additional aliquot of soil without any methanol or preservative must be collected for use in determining percent moisture (i.e., for dry weight determination).

8.3.6 Upon the return to the laboratory, the sample vials (now containing both the Methanol/Surrogate solution and approximately 10g of soil) are reweighed using the barcode reader and analytical balance. Record this weight (Weight 2) in the methanol tracking system. Calculate the initial weight of soil by subtracting Weight 1 from Weight 2.

8.3.7 All samples for volatile analysis are protected from light and stored at 4°C±2°C in an area free of organic solvents. No standards or solvents are stored in the sample refrigerators or in the surrounding area.

8.3.8 All samples preserved in this manner must be analyzed within 14 days of collection.

9.0 Quality Control

9.1 Specific quality control procedures are outlined in each of the determinative methods that follow this purge and trap technique of sample introduction. Standard quality assurance practices are used with all methods.

9.2 Reagent water blanks are analyzed to ensure that reagents and/or sample dilutions are free of interferences.

9.3 Method blanks are prepared and analyzed in the same manner as samples, and are carried through the entire analytical procedure to show that each system is in control.

9.4 Potential cross-contamination of samples stored in lab refrigerators is monitored by preparation, analysis and evaluation of storage blanks. Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After one week, the storage blanks are removed and analyzed. Additional details can be found in TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

9.5 An initial demonstration of accuracy and precision is performed for each determinative method. This demonstration is done for each sample introduction technique.

10.0 Procedure

10.1 This section provides guidance for the analysis of low level soils using a closed system purge and trap procedure. Reference the SOPs for the determinative methods for additional details as applicable: TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) or ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*).

10.2 Instrument operating parameters are set at the beginning of a method of analysis and remain constant throughout the entire analytical procedure. Again, reference the SOP for the applicable determinative method for specifics. Sample operating conditions are listed here:

Example Instrument Operating Parameters

Purge and trap unit	
Purge Time	11 minutes
Dry Purge	1 Minutes
Purge Gas	Helium
Purge Flow	40-45 ml/min
Purge Temp	Water - Ambient; Solids - 40^{0}C
Trapping Temp	Ambient, <30^{0}C
Desorb Time	1-2 Minutes
Desorb Temp	Vocarb - 260^{0}C
Gas chromatograph	
Injector	180^{0}C
Carrier Gas	Helium
Carrier Flow	6 ml/min
Oven Program	35 - 250^{0}C
Run Time	30 Minutes
Mass Spectrometer	
Electron Energy	70 volts (nominal)
Mass range	35-260 AMU
Scan time	0.9 sec./scan
Source Temp	200^{0}C
Separator Temp	180^{0}C

10.3 All samples are screened prior to analysis, using the procedure outlined in TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID SW846 Method 5021(modified),* current revision. In addition to reducing excessive instrument contamination and/or extensive instrument clean up, screen data provides the analyst with an approximate dilution factor and is used to determine which sample preparation technique should be used for a particular sample. If the estimated concentration from the screening procedure shows concentrations below 200 ug/kg, follow the low concentration closed system purge and trap method. If the screen result shows concentrations >200 ug/kg, follow the medium level procedure.

10.4 Low Level Soil Procedure

10.4.1 Low level soils are field collected as described in Section 8.1 using 40 mL VOA vials with PTFE-faced septa (closed system purge and trap), En Core® Samplers, Terra Core® kits or bulk soil.

10.4.2 Set up the purge and trap autosampler (Archon 5100A or EST Centurion, see Section 6.1) for the closed system method for low-level soils. The purge and trap autosampler will add 5 mL reagent water, and 1uL of the internal standard and/or surrogate spiking solution to the sample vials. The heating block will heat each sample to 40° C and hold for 0.5 minutes before purging begins.

10.4.3 Tuning, calibration, and method blank analysis:

- Tuning and calibration is accomplished as detailed in the SOP for the applicable determinative method.

- The volume of water used when purging a calibration standard must be the same as the volume of water purged during a sample analysis. Since the low-level soils have 5 mL water added at vial preparation plus the 5 mL of reagent water added by the purge and trap autosampler prior to purging, a total volume of 10 mL is required for calibration standards.

- Internal standard (if required by the determinative method) is added to each calibration standard by the purge and trap autosampler, in the same manner it will be added to the samples.

- After successful calibration, analyze a method blank. The method blank must meet all criteria in the determinative method. A successful method blank shows that the system is free of interferences and sample analysis may begin.

10.5 Medium Level Soil Procedure

10.5.1 Medium level soils are typically field collected using laboratory prepared 40 ml VOA vials (with PTFE-faced septa) containing 25 mL of the Methanol/Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed).

10.5.2 When preparing a medium level soil from a bulk soil sample, do not decant any supernatant liquids. Quickly mix the contents of the sample container with a narrow spatula. Weigh out 5g of soil into a 40ml vial, add 10ml of Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) and cap the container and proceed with Section 10.5.6.

10.5.3 For samples collected in the Encore sampling device, break open one end and push the aliquot of soil out into a vial. Add 10ml of the Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) and cap the vial and proceed with Section 10.5.6.

10.5.4 For Method 8260 samples collected as Terra Cores® in 5 mL or 10 mL methanol add 5 uL and 10 uL of the 2500 ppm 8260 Methanol/Surrogate mix (see Section 7.1.4) respectively to extract and proceed with Section 10.5.6.

10.5.5 For Method 8015 (GRO) samples collected as Terra Cores® in 5 mL or 10 mL methanol add 2.5 uL and 5 uL of the Methanol/Surrogate Mix surrogate solution (see Section 7.1.5) respectively and proceed with Section 10.5.6.

10.5.6 Cap and shake the extract for 2 minutes. Allow soil to settle.

10.5.7 Transfer 2 mL of the extract to a 2 mL amber glass vial for storage with minimal headspace.

10.5.8 Refrigerate at 4°C until the time of analysis.

10.5.9 Analyze a portion of this extract using Method 5030 purge and trap for aqueous samples (TestAmerica Edison SOP No. ED-MSV-001, *Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*). The appropriate dilution factor is calculated from the low-level analysis.

10.6 High Level Soil Procedure

10.6.1 High level soils have been preserved in the field by sampling either 10 g of soil into vials containing 25 mL Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) or 5 g of soil into vials containing 10 mL Methanol/Surrogate mix solution (again, either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed).

10.6.2 Screen high level soils by Method 5021 to determine the proper dilution factor prior to analysis (see TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics,Static Headspace with GC FID SW846 Method 5021(modified),* current revision.)

10.6.3 Transfer 2 ml of the extract to a 2 mL amber glass vial for storage with minimal headspace.

10.6.4 Analyze a portion of this extract using Method 5030 purge and trap for aqueous samples.

10.7 Sample Analysis

10.7.1 Remove sample vials from storage and allow thawing to ambient temperature before analysis. Shake the vial gently to ensure that the contents move freely within the vial. Place the sample vials in the autosampler tray.

10.7.2 Schedule the autosamplers to run the samples using the low level soil method. 5 mL of reagent water plus 1 uL of internal standard/ surrogate spiking solution is added to the sample vial directly through the septum.

10.7.3 Prior to purging, the sample is heated to 40°C for 0.5 minutes.

10.7.4 Purge the sample with helium for 11 minutes while agitating with magnetic stirring.

10.7.5 The purgeable compounds will flow out of the vial through a glass lined transfer line to a sorbent trap.

10.7.6 Desorb the contents of the trap on to the GC column by rapidly heating the trap to 260 °C and backflushing it with helium.

10.7.7 Begin the GC temperature program and data acquisition.

10.7.8 Re-condition the trap after desorb by baking at 260° C for 8-12 minutes.

10.7.9 Perform quantitative and qualitative analysis using TARGET software. Evaluate data according to each determinative method.

10.7.10 If a re-analysis is required, perform the re-analysis using the second frozen sample vial.

10.7.11 If a dilution is required, analyze the methanol preserved aliquot by Method 5030.

10.7.12 Technical acceptance criteria for sample analysis. (Note: these are general guidelines; please see the SOP for the determinative method for more specific acceptance criteria)

- The samples must be analyzed on a GC or GC/MS system meeting the initial calibration, continuing calibration and blank technical acceptance criteria.
- The sample must be analyzed within the required holding time.
- The sample must have an associated method blank meeting the blank technical acceptance criteria.
- The percent recovery of each of the system monitoring compounds in the sample must be within the acceptance windows.
- After analyzing a sample that exceeds the initial calibration range the analyst must either analyze an instrument blank (using the same purge inlet if using an auto sampler) which must meet technical acceptance criteria for blank analysis or monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample that exceeded the calibration range.

10.7.13 Corrective Action for Sample Analysis (Note: these are general guidelines; please see the SOP for the determinative method for more specific corrective actions):

- Samples must meet technical acceptance criteria before reporting data.

- Corrective action for failure to meet instrument performance checks, initial, continuing calibration and method blanks must be completed prior to sample analysis.

- Corrective action for system monitoring compounds and internal standard compounds that fail to meet acceptance criteria must be completed prior to sample analysis.

- If any of the system monitoring compounds and internal standard compounds fail to meet acceptance criteria, check all calculations, instrument logs, the system monitoring compound and internal standard compound spiking solutions and the instrument operation. If the calculations were incorrect, correct calculations and verify that the system monitoring compound recoveries and internal standard compound responses meet acceptance criteria.

- Check the preparation of the internal standards and system monitoring compounds for concentration and expiration.

- Verify that the instrument is operating correctly.

- Data that fails to meet minimum acceptance criteria will be annotated (flagged) with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Reports will be initiated in order to provide for investigation and follow-up.

11.0 Calculations / Data Reduction

11.1 Methanol volume correction for soil moisture content (medium/high level methods):

$$Vt\ (\mu L\ solvent/water) = [\ ml\ of\ solvent + \frac{(\%moisture \times g\ of\ sample)}{100}] \times 1000\ uL/mL$$

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training* (latest revision) for the laboratory's training program.

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out.

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system**.**

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These

boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

Teris Profile Number (incineration): 50016710
Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

- Returned Methanol Preservative - Methanol preserved vials are collected in satellite accumulation and then transferred to 55 gallon open top steel waste drums in the waste room. These waste drums are then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, *Method 5035A: Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 0, December 1996.

15.2 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3 TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics,Static Headspace with GC FID SW846 Method 5021(modified),* current revision.

15.4 TestAmerica Edison SOP No. ED-MSV-001, *Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*.

15.5 TestAmerica Edison SOP Nos. ED-GCV-006, *Gasoline Range Organics using GC FID Method SW846 8015, current revision.*

15.6 TestAmerica Edison SOP No ED-MSV-005, *SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision.*

15.7 United States Environmental Protection Agency, *Method 8000C: Determinative Chromatographic Determinations*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, March 2003

15.8 TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

15.9 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001)

15.10 TestAmerica SOP No. ED-GEN-022, *Training* ,latest revision.

15.11 TestAmerica Edison SOPs Nos. ED-SPM-007, *Disposal of Samples and Associated Laboratory Waste, current revision.*

15.12 TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures,* current revision.

16.0 Method Modifications:

16.1 N/A

17.0 Attachments

17.1 N/A

18.0 Revision History

- Revision 10, effective date: 4/11/2019
  - Section 8.2.1: Added temperature preservation requirement for Medium level extracts.

- Revision 9, effective date: 11/27/2012
  - Updated referenced Lab Quality Manual section and appendix numbers as appropriate

- Revision 8, effective date: 12/6/2010
  - Section 3: revised to reference new location for definitions.
  - Sections 8.2.1.4 and 8.3.9 referencing methanol solvent adjustment for solid samples with significant moisture content (>10%) was removed.

- Revision 7, effective date: 11/4/2008:
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Updated the SOP to comply with SW846 Method 5035A (was 5035). Retitled SOP accordingly.
  - Section 1.1.5: Added reference to TestAmerica Edison SOP No. ED-MSV-001(*Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*).
  - Section 1.1.6: Added references to TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current*

revision) and ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*)

- Section 1.1.7: Added reference to Quality Assurance Manual for method modifications.
- Section 3: revised to reference new location for definitions.
- Section 4: revised to include a reference to storage blanks and the applicable SOP: TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination*, current revision.
- Section 5 (Safety): Revised to include most up to date corporate health and safety references and information.
- Section 6: Updated with current instrumentation and configurations.
- Section 7.0: added details of the solvent testing and approval program.
- Section 7.2: Updated standards sources and catalog numbers.
- Section 8: Updated with additional details for each type of preservation/analysis (low level, medium level, high level). Added used of Terra Core® Sampling Kits (Section 8.1.3).
- Section 8: Updated to include requirement to adjust the methanol volume (medium/high level) for soil moisture content.
- Section 9.0: Added details of storage blanks and reference to TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision..
- Section 10: Revised and expanded to include instrument operating conditions and specific sample prep details for low level, medium and high level preps.
- Section 11: Deleted reference to SOP for Organic Calculations. Added statement that calculations can now be found in the applicable determinative methods.
- Section12.0: added reference to Training SOP.
- Section 13.0: Revised to include most up to date company policy on Pollution Control as well as to include corporate health and safety references and information.
- Section 14.0: Revised to include most up to date company policy on Waste Management as well as to include corporate health and safety references and information.
- References: Expanded to include more specific SOP references.
- Revision history: updated.

Title: SW846 Method SW8081B, Analysis of Organochlorine Pesticides by Gas Chromatography

Approvals (Signature/Date):

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

Method 8081B is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, using dual fused-silica, open-tubular, capillary columns with electron capture detectors (ECD). The list of analytes and their corresponding reporting limits are as follows:

Parameter		Soil	Water	Leachate
	CAS Registry No.	Reporting Limits (ug/Kg)	Reporting Limits (ug/L)	Reporting Limits (mg/L)
Aldrin	309-00-2	6.7	0.02	---
Alpha-BHC	319-84-6	2.0	0.02	---
Beta-BHC	319-85-7	2.0	0.02	---
Delta-BHC	319-86-8	2.0	0.02	---
Gamma-BHC (Lindane)	58-89-9	6.7	0.02	0.00050
Chlordane	57-74-9	67	0.50	0.0050
4,4' –DDD	72-54-8	6.7	0.02	---
4,4' –DDE	72-55-9	6.7	0.02	---
4.4' –DDT	50-29-3	6.7	0.02	---
Dieldrin	60-57-1	2.0	0.02	---
Endosulfan I	959-98-8	6.7	0.02	---
Endosulfan II	33213-65-9	6.7	0.02	---
Endosulfan sulfate	1031-07-8	6.7	0.02	---
Endrin	72-20-8	6.7	0.02	0.00050
Endrin aldehyde	7421-93-4	6.7	0.02	---
Endrin ketone	53494-70-5	6.7	0.02	---
Heptachlor	76-44-8	6.7	0.02	0.00050
Heptachlor epoxide	1024-57-3	6.7	0.02	0.00050
Methoxychlor	72-42-5	6.7	0.02	0.00050
Toxaphene	8001-35-2	67	.50	0.0050
Gamma- Chlordane	5103-74-2	6.7	0.02	---
Alpha-Chlordane	5103-71-9	6.7	0.02	---
Mirex	2385-85-5	6.7	0.010	---

The most current MDLs and RLs for this method can be found in the active Eurofins LIMS (TALS) SW846 8081B Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Samples undergo a preparation step prior to analysis by SW846 Method 8081B. A measured volume or weight of sample (15g for soil, 1 g for waste, 250 ml for water, and 250 ml for TCLP) is extracted using the appropriate matrix-specific sample extraction technique. (Reference the applicable Organic Sample Prep SOPs listed below). The effective final volume is usually between 5 and 20 ml in hexane.

2.1.1. Aqueous samples are extracted using SW846 Method 3510C (SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel*).

2.1.2. Solid samples are extracted using SW846 Method 3550B: Sonication (*SOP No. ED-ORP-018: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction*) or SW846 Method 3546: Microwave (SOP No. ED-ORP-044: *Procedure for the Microwave Extraction of Solids,SW846 3546).*

2.1.3. Organic liquids are prepared using SW846 Method 3580A (*SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs*).

2.1.4. Extract cleanup steps are employed depending on the nature of the matrix interferences. Suggested cleanups include SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*) and SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*).

2.2. After cleanup, a small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to an Electron Capture Detector (ECD) which is used to detect the compounds eluting from the GC.. Quantitation is accomplished by comparing the area response of each target analyte relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8081B.

2.3. For pesticide analysis a system performance check (DDT/Endrin breakdown) and a calibration verification standard must be run prior to analysis. Failure of either will generally indicate the need for injection port/column maintenance and/or recalibration.

2.4. Samples are analyzed after all the necessary checks have been performed. Samples analyzed for pesticides require an additional post analysis Quant Report to be printed and attached to the chromatographic report.

2.5. All samples are then manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

4.1.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. If sulfur is encountered, employ the sulfur removal procedures detailed in SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*). Note that the recover of Endrin Aldehyde is adversely affected by the TBA cleanup procedure detailed in this method. Accordingly, this compound must be determined prior to sulfur cleanup.

4.3. Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*).

4.3.1. Check Florisil prior to use to assure quantitative recovery of targeted analytes. Duplicate checks are required for each new lot or every three hundred samples whichever is more frequent.

4.3.2. Check Florisil by spiking 1ml of the Pest Std Mix A midpoint (Supelco Catalog No. 47977) and 0.5 ml of trichlorophenol (Absolute Standardsl Catalog No. 20024) onto the cartridge and concentrating to final volume of 1 ml. Inject 1 ul onto a capillary column, conducting the elution and analyzing the extract. Recovery is acceptable if all pesticides are recovered at 80 - 110% and the recovery of trichlorophenol is <5% and co-eluting interfering peaks are absent from the extract.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation:

6.1.1. Gas Chromatograph: The system used is an HP and an Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC). Each GC is equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides. All operations are as automated as possible with the equipment utilized.

6.1.2. Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles samples between the sample tray and the injector turret.

6.1.2.1. The samples are injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.

6.1.2.2. Liners: The injection port is each fitted with replaceable, heavy-walled siltek-coated glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.

6.1.2.3. Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35°C and 350°C.

6.1.2.3.1. Two dissimilar columns are used for analysis. A Restek RtxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column is used for sample quantitation. The secondary confirmation column is a Restek RtxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column.

6.1.2.4. Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.

6.1.2.4.1. Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas (P-5 & Nitrogen) is fed from a supply other than the injection port.

7. Reagents and Standards

7.1. Reagents

7.1.1. Gases: Hydrogen is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. P-5 and Nitrogen) is used as make-up gas. It is introduced to the GC via the make-up gas adapter at the end of the capillary column. Hydrogen is supplied via a Parker Balston H2 Generator. Nitrogen & P-5 is supplied by Air Gas

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the GC The traps are as follows:

7.1.1.1.1. Hydrocarbon trap
7.1.1.1.2. H_2O trap
7.1.1.1.3. O_2 scrubber

7.1.1.2. Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).

7.1.2. Solvents used in the extraction and cleanup procedures include n-hexane, methylene chloride, and acetone that are exchanged to n-hexane prior to analysis.

7.1.3. Hexane is required in this procedure. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2. Standards

7.2.1. Standards are purchased as concentrated solutions (see Section 7.2.2).

NOTE: Independent sources are used for quantitation standards and spiking standards

7.2.1.1. Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.2.2.1.

7.2.2. Standard mixes and sources *.

Standard Name	Source
Organochlorine Pesticide Mix AB #3	Restek Catalog No. 32415
Organochlorine Pesticide Mix AB #3.sec	Restek Catalog No. 32415.sec (second source)
Pesticide Surrogate Mix	Restek Catalog No. 32000
Endrin/DDT	Supelco Catalog No 48282
Chlordane	Restek Catalog No. 32021
Toxaphene	Restek Catalog No. 32005
Toxaphene (different pattern)	Restek Catalog No. 32071
Mirex	Accustandard Cat. No. P-066S-10X
1-Bromo-2-nitrobenzene (internal standard)	Restek Cat. No. 32279

*Suppliers with equivalent standards may be substituted..

The components of each standard mix are as follows (note: 'sec' indicates second source):

Parameter	Supplier	Catalog No.	Concentration of Standard (ug/ml)
Aldrin	Restek	32415 & 32415.sec	2000
Alpha-BHC	Restek	32415 & 32415.sec	2000
Beta-BHC	Restek	32415 & 32415.sec	2000
Delta-BHC	Restek	32415 & 32415.sec	2000
Gamma-BHC (Lindane)	Restek	32415 & 32415.sec	2000
Alpha -Chlordane	Restek	32415 & 32415.sec	2000
Gamma -Chlordane	Restek	32415 & 32415.sec	2000
Technical Chlordane	Restek	32021	1000
4,4' –DDD	Restek	32415 & 32415.sec	2000
4,4' –DDE	Restek	32415 & 32415.sec	2000
4.4' –DDT	Restek	32415 & 32415.sec	2000
Dieldrin	Restek	32415 & 32415.sec	2000
Endosulfan I	Restek	32415 & 32415.sec	2000
Endosulfan II	Restek	32415 & 32415.sec	2000
Endosulfan sulfate	Restek	32415 & 32415.sec	2000
Endrin	Restek	32415 & 32415.sec	2000
Endrin aldehyde	Restek	32415 & 32415.sec	2000
Endrin ketone	Restek	32415 & 32415.sec	2000
Heptachlor	Restek	32415 & 32415.sec	2000
Heptachlor epoxide	Restek	32415 & 32415.sec	2000
Methoxychlor	Restek	32415 & 32415.sec	2000
Toxaphene	Restek	32005	1000
Toxaphene	Restek	32071	5000
Mirex	Accustandard	861428-U	1000
4,4-DDT	Supelco	18282	500
Endrin	Supelco	48282	500
Decachlorobiphenyl (DCB)	Restek	32000	200
Tetrachloro-m-xylene (TCmX)	Restek	32000	200
1-Bromo-2-nitrobenzene (internal standard)	Restek	32279	1000

7.2.2.1. Standards Preparation

7.2.2.1.1. Calibration Mix (Organochlorine Pesticide Mix)

The 5 point calibration standards are prepared as detailed in the following table using volumetric glassware and hexane as the diluent:

Initial Calibration Standards Prep (Organochlorine Pesticide Mix)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Organochlorine Mix AB#3 ***(2000 ug/ml)*** *Restek (Cat No..32415)in Hexane:Toluene*	Volume brought up to 200 ml hexane: *0.25 ul*	Volume brought up to 200 ml hexane: *5.0 ul*	Volume brought up to 500 ml hexane: *25 ul*	Volume brought up to 200 ml hexane: *25 ul*	Volume brought up to 200 ml hexane: *50 ul*
Initial Calibration Standards Prep (TCMX/DCB Surrogate Mix)					
Stock Std	**25 ppb***	**50 ppb**	**100 ppb**	**150 ppb**	**200 ppb**
Tetrachloro-m-xylene/ Decachlorobiphenyl Surrogates Mix (200 ug/ml) *Restek (Cat, No, 32000) in Acetone*	Volume brought up to 200 ml hexane: *6.25 ul*	Volume brought up to 200 ml hexane: *50 ul*	Volume brought up to 500 ml hexane: *250 ul*	Volume brought up to 200 ml hexane: *150 ul*	Volume brought up to 200 ml hexane: *200 ul*
Initial Calibration Standards Prep (Mirex)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Mirex ***(1000 ug/ml)*** *Accustandard (Cat. No. P-066S-10x) in Methanol*	20x dilution of 50 ppb	10x dilution of 500 ppb	5x dilution of 500 ppb	2x dilution of 500 ppb	Volume brought up to 100 ml hexane: *50 ul*

Note: 20 ul of 5 ug/ml Internal Standard solution is added to all calibration standards prior to analysis.

7.2.2.2. **Pesticide Surrogate Spike Mix (10 ug/ml**) : The Pesticide 10ug/ml surrogate spiking solution is prepared by diluting 10 ml of 200 ug/ml of the Pesticide Surrogate Mix (Restek-32000) in to 200 ml of Acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.3. **Pesticide Surrogate Spike Mix (2 ug/ml) for the reduced volume extraction option:** prepare a 2 ug/ml spiking solution by diluting 10 ml of the 10 ug/ml stock standard described in 7.2.2.2 to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.4. **Pesticide Internal Standard Spike Mix (5 ug/ml):** The Pesticide 5 ug/ml internal standard spike mix is prepared by

dilution 1 ml of 1000 ug/ml of the 1-Bromo-2-Nitrobenzene standard (Restek 32279) in to 200 ml of Hexane. 20 ul of this solution is added to all standards, QC samples and field sample extracts prior to analysis.

7.2.2.5. **Pesticide Spiking Standard (20 ug/ml):** The Pesticide Spiking Mix containing the single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul of the Organochlorine Pesticide Mix AB#3 (Restek 32415 to a 50 ml final volume with acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.6. **Pesticide Spiking Standard (4 ug/ml) for the reduced volume extraction option**: prepare a 4 ug/ml spiking solution by diluting 10 ml of the 20ug/ml standard prepared in Section 7.2.2.4 above to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.7. **System Performance Solution (Breakdown Check)** 3,3-DDT and Endrin at 0.25 ug/ml): The breakdown check is prepared by taking 250 ul of 500 ug/ml DDT/Endrin Mix (Supelco Catalog No 48282) and bringing it up to a volume of 500 ml with hexane

7.2.2.8. **Technical Chlordane Calibration Solution** (1.0 ug/ml solution w/ surrogates TCMX & DCB at 0.10 ug/ml): 100 ul of 1000 ug/ml Technical Chlordane Standard (Restek Catalog No 32021) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. ***NOTE:*** ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.9. **Toxaphene Calibration Solution** (1.0 ug/ml solution w/surrogates TCMX & DCB at 0.1 ug/ml) : 20 ul of 5000 ug/ml Toxaphene stock (Restek Catalog No 32071) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. ***NOTE:*** ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.10. **Initial Calibration Verification (ICV)** Preparation: follow the instructions above for midpoint standards substituting the second source standard (catalog no. suffix = sec) for the primary standards.

8. Sample Collection, Preservation, Shipment and Storage

8.1. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

8.2. Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 250 ml	250 ml	Cool 4 ± 2ºC	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 ± 2ºC	14 days to extraction; Analyze within 40 days of extraction	SW846

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standard [1]	Every sample[3]	Response within -50% to +100% of most recent cal standard

LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method Blanks** are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall

below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The recoveries of the LCS must fall within lab generated acceptance criteria (refer to the current TALS Method Limit Group database). If the LCS recovery results are outside of these limits, the extract is reanalyzed. If LCS recoveries are still outside of QC limits after extract reanalysis but recoveries for the Matrix Spike/ Matrix spike Duplicate (MS/MSD) are within QC limits, the data is reported and a Non Conformance Memo (NCM) is written.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to the current TALS Method Limit Group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated. If the LCS recoveries meet criteria the data is reported and a Non-Conformance Memo (NCM) is written.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to the current TALS Method Limit Group database*)*. Minimum requirements for surrogate evaluation:

- Both surrogates must have reportable results that meet the acceptance criteria;
- Reported surrogates must be from a column with a passing CCV;
- At least one surrogate must pass on any column from which target analytes are identified and reported.

If both TCMX and DCB recovery are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.1.5. Internal Standard: The internal standard (1-bromo-2-nitrobenzene) must elute within 30 seconds of and have an area response of 50 to 100% as compared to the most recent preceding calibration standard.

9.1.6. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification: The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to

demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

9.2. Instrument QC

9.2.1. GC System Performance Check

9.2.1.1. **Endrin/4,4'-DDT Breakdown:** Prior to performing any standards or sample analysis, a daily check is made on the chromatographic performance of the system. This performance check is made by injecting a standard of Endrin and DDT, each at a 250-ppb level (see Section 7.2), and calculating the percentage breakdown for each compound.

9.2.1.2. Ideally, only two peaks will be seen (one for Endrin and one for DDT). As a rule, this is not the case. It is normal to observe up to six peaks. Three peaks are attributable to Endrin and its degradation products: Endrin Aldehyde (EA) and Endrin Ketone (EK). Three peaks are attributable to DDT and its degradation products: DDE and DDD. Calculate the percentage breakdown as follows:

Endrin:

$$\frac{\text{(Areas of EA + EK)}}{\text{(Areas of EA + EK + Endrin)}} \times 100 = \%\ \text{breakdown Endrin}$$

DDT:

$$\frac{\text{(Areas of DDE + DDD)}}{\text{(Areas of DDE + DDD + DDT)}} \times 100 = \%\ \text{breakdown DDT}$$

9.2.1.3. If the percentage breakdown for either Endrin or DDT is greater than 15%, the system CANNOT be used for pesticide analysis. If the Endrin/DDT performance check fails, injection port/column maintenance must be performed. Usually, changing the glass wool/liner will cure most breakdown problems in the injection port. Depending upon the nature of the samples, the entire injection port will occasionally need to be cleaned. This cleaning is best done with 1:1 Acetone: Hexane. Another routine maintenance operation to improve column performance is the removal of the

first 3 cm of the column. (Note: the septa should be changed each time the injection port is opened).

9.2.1.4. After injection port/column maintenance has been performed, and the columns have been given time to equilibrate (baseline back down to normal) the Endrin/DDT must be re-injected and the system performance re-evaluated.

9.2.2. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.2.1. ***Initial Calibration Range*:** Single component pesticides are calibrated using a five-point calibration range. Multi-component pesticides are calibrated using a single point calibration at the anticipated midpoint of the calibration range. Standards are prepared following the instructions in Section 7.2.

9.2.2.2. Single response Pesticide Calibration: All single component pesticides and two surrogates are calibrated with a minimum of 5 concentrations. Single component pesticides are analyzed at 10, (5 ppb for the 125 ml initial volume method) 5, 100, 250 and 500 ppb. Surrogate standards are analyzed at 25, 50, 100, 150 and 200 ppb. See Section 7.2 for details on standard prep.

9.2.2.3. Multi-response Pesticide Calibration: Chlordane (technical) and Toxaphene initial calibration is accomplished by analysis of a single point at 1000 ppb (see Section 7.2 for details on standard prep).

9.2.2.4. ***Initial Calibration Verification (ICV):*** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2 and must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3. Continuing Calibration Verification (CCV): For single component pesticides, a mid-point Continuing Calibration Verification (CCV) must be analyzed every 12-hours or 20 samples (whichever is more frequent). 'Samples' here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… For multi-response pesticides a CCV must be analyzed within 12 hours of any multi-response pesticide detects.

9.2.3.1 Analysis of Replicate CCVs: Occasionally dual, sequential CCVs may (for a variety of reasons) be included in an analytical sequence. When such replicate CCVs are injected both must be evaluated as detailed in the table below (reference Eurofins Document CA-Q-W-008, 'Technical Guidance on the Use and

Evaluation of Replicate Continuing Calibration Verification (CCV)').

Dual CCV Evaluation Decision Tree		
Inejection	**QC Acceptance**	**Action**
CCV1 **CCV2**	**Pass** **Pass**	Continue analytical sequence. Data acceptable based on calibration.
CCV1 **CCV2**	**Pass** **Fail**	Analyses before CCV1 may be accepted. Re-analyze all samples that were analyzed after failed CCV2.
CCV1 **CCV2**	**Fail** **Pass**	Re-analyzed all samples that were analyzed before CCV1 and after the previous compliant CCV. Analyses after CCV2 may continue.
CCV1 **CCV2**	**Fail** **Fail**	Re-analyze all samples since the last acceptable CCV. Perform maintenance and/or recalibration prior to re-analysis.

9.2.3.2 In the event that replicate CCVs are included in a sample injection sequence, the acceptance of the associated sample analyses must be properly evaluated and the evaluation process must be documented. Under no circumstances is it allowable to accept sample data based on the evaluation of only 1 of the CCV replicates.

9.2.3.3 Clear documentation of the evaluation of the CCV must be included in the analytical sequence run log (i.e., 'Pass' or 'Fail' in the comment section for each CCV). Additionally, clear documentation of corrective action taken in the event of CCV failures must be included in the run log (see table above for actions that need documentation).

Note: It is acceptable to use the CCV solution as a primer at the beginning of an analytical sequence however the injection should be documented as a 'Primer' in the sequence rather than as a CCV.

9.2.4. Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability (for more detail on the following procedures refer to

Eurofins Corporate Technical Services Policy No. CA-T-P-005, “Policy for Determining RT Windows for GC/ECD Tests” and Eurofins Corporate Technical Services Work Instruction No. CA-T-WI-010, “Further Guidance on the RT Window Policy No. CA-T-P-005”).

9.2.4.1.1. Initial determination of RT windows.

9.2.4.1.1.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration of the first CCV in the daily sequence, whichever is most recent.

9.2.4.1.1.2. Use a default RT window of 0.01 minutes. At the end of the batch evaluate all CCVs and LCSs in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.

9.2.4.1.1.3. Multiply the maximum deviation by 1.5. This is the retention time window, unless the result is less than 0.01 minutes, in which case the window is set at 0.01 minutes. For example, if the maximum RT deviation for a specific analyte is 0.008 minutes, then the RT window is set at ±0.012 minutes.

NOTE: For the multi-component analytes, for example Aroclors, Toxaphene and Technical Chlordane, the maximum deviation must be evaluated for each of the 3 to 6 major peaks used for sample calculations.

9.2.4.1.1.4. Retention time windows for analytes of interest must not overlap for GC analysis.

9.2.4.1.2. Ongoing evaluation of retention time windows

9.2.4.1.2.1. Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below).

9.2.4.1.2.2. Matrix spike analytes may fall outside of the retention time window if there is a large non-target peak coeluting with the analyte in the matrix spike.

9.2.4.1.2.3. If any analytes fall outside of the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

9.2.4.1.2.4. Retention time windows should be reliably narrower than ±0.03 minutes. If RT windows wider that this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained.

9.2.4.2. **Initial Calibration Range:** Internal standard calibration is employed for this method. A response factor is calculated for each analyte at each calibration concentration.

Response factor = $((A_x)\ (C_{is}))/((A_{is})\ (C_x))$

Where:
A_x = area of the compound
C_x = Concentration of the compound
A_{is} = area of the internal standard
C_{is} = Concentration of the internal standard

9.2.4.2.1. Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

Where:
RF = Response Factor

9.2.4.2.2. If the % RSD across the 5 point range is <20% for any given compound the calibration can be assumed to be linear and the average response factor can be used to calculate concentrations of target compounds in samples.

9.2.4.2.3. If the % RSD is >20% for any given compound, a first order linear regression may be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be $\geq$ 0.990 for the calibration to be acceptable. Calibration is checked every

12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single component pesticide standards.

9.2.4.2.4. Chlordane and Toxaphene Calibration: Chlordane and Toxaphene are multiple response pesticides and are calibrated with a minimum of 5 points as required (i.e, within 12 hours of either analyte being detected in a sample). Three to eight peaks are used for calculation of response factors and the same criteria detailed above is applied to determine acceptability of calibration.

9.2.4.2.5. Resolution: All single component analyte peaks must exhibit at least 80% chromatographic resolution. The analyst performs a visual check of the mid-level initial calibration standard and all subsequent calibration checks (ICV/CCV). If the resolution requirement is not met instrument maintenance should be performed followed by re-calibration. Percent resolution can be calculated when necessary as follows:

$$\% \text{ Resolution} = V/H \times 100$$

Where:

V= Depth of the valley between two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks

H = Height of the shorter of the adjacent peaks

9.2.4.3. **Relative Standard Error:** The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation. The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r_2) for

testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on *r.* As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point

9.2.4.4. Initial Calibration Verification (ICV): An ICV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.2.4. The calculated concentration of the ICV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5)

9.2.4.5. Continuing Calibration Verification (CCV): A CCV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.3.. The calculated concentration of the CCV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 20% after a single CCV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5).

Step	***Standards***	***Type***	***Control Limit***	***Frequency***
Method # 8081B				
GC System Performance Check	*Endrin/DDT, 250 ppb*	*Performance check*	*15% breakdown*	*At beginning of 12 hour clock and after system maintenance*
*Initial Calibration**	2.5, 50, 100, 250 and 500 ppb for single response, 1000 ppb for multi response	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r ≥0.990 or <20%RSE*	*As required when ICV or CCV do not meet requirements*
ICV	*100 ppb*	*Average*	*± 20%D*	*Once after each initial calibration*
CCV	*100 ppb*	*Average*	*± 20%D*	*Every 12 hrs or 20 samples, whichever is more frequent*

10. Procedure

10.1. Gas Chromatograph Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. General Operating Conditions

10.1.2.1. Injection System: A split/splitless injection port with electronic pressure control (EPC) is used. Thirty seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

10.1.2.2. The EPC is used in the ramp pressure mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	Rate	Final Pressure	Hold
12 psi	2.5 min	7 psi/min	4 psi	1.50 min
		5 psi/min	9 psi	1.40 min
		9 psi/min	13 psi	2.00 min

10.1.2.3. For pesticide analysis the normal operating conditions of the injection port are as follows:

Injection port Temperature:	250°C
Column flow:	12.3 ml/minute
Split vent flow:	5 ml/minute
EPC:	Pressure Ramp
Detector temperature	330C

10.1.2.4. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. The plug of glass wool is located in the liner between the double goose neck.

10.1.2.5. This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.6. The glass wool will be changed when changing the liner. The changing of the glass wool/liner is based upon the breakdown of

an Endrin/DDT standard. This is covered in further detail in section 10.2.1.

10.1.2.7. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.8. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.9. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.10. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.2.11. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.2.11.1. A standard oven program for pesticide analysis is employed for all columns as follows:

Initial Temp	**Hold Time1**	**Rate1**	**Temp1**
160°C	0.62 min	30°/min	244°C
Hold Time2	**Rate 2**	**Final Temp**	**Final Time**
2.5min	21°/min	315°C	3.0min

10.1.2.12. Detectors: Detectors operate at 330°C and need to be supplied with 60 ml/min total flow. They are essentially maintenance free

on a day-to-day basis. They are routinely baked out at 330°C to remove persistent contaminants. On occasion the detectors may be baked out at a higher temperature to remove contaminants with an extremely high boiling point (CAUTION: Do not exceed the maximum detector temperature of 380°C).

10.1.2.12.1. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.2.13. Chemstation: HP Chemstation software is used for automation of runs and data acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.2.14. Eurofins Chrom data processing software is used for the processing of the chromatography data files. Calibrations, verification standards and samples are processed and reviewed using this database. Chrom is integral to Eurofins LIMS (TALS) which is used to generate all reports..

10.2. Analytical Sequence

10.2.1. The analytical sequence for performance checks, initial calibration, calibration verifications and sample analysis is described in the following sections.

10.2.2. Before calibration standards are analyzed the GC Performance Check Standard (see Section 9.2.2.1) must first be analyzed and evaluated to check the performance of the injection port and column with regard to catalytic active sites. The breakdown for both Endrin and DDT in the Performance Check Standard must be less than 15%. If the performance check fails this criteria system maintenance must be performed and the check successfully reanalyzed before proceeding with calibration.

10.2.3. A five point initial calibration (ICAL) is analyzed and evaluated for each of the 21 single response pesticides plus surrogates as described in Section 9.2.4.2. When needed a 5 point ICAL is also analyzed for Mirex.

10.2.4. Calibration for Technical Chlordane and Toxaphene: Chlordane and Toxaphene are multiple response pesticides containing at least 3-8 primary peaks each. A single point calibration check standard at a concentration of

1000ug/l is analyzed for Chlordane and Toxaphene. A full 5 point calibration range is analyzed for these compounds should they be detected in client samples..

10.2.5. A second source initial calibration verification (ICV) is analyzed and evaluated for each of the 21 single response pesticides as described in Section 9.2.4.3. When needed an ICV is also analyzed for Mirex. **NOTE**: The 12 hour time clock for Pesticides commences with the injection of the first Pesticide Calibration Standard or Verification.

10.2.6. GC Performance Check Standard is analyzed and the breakdown of Endrin/DDT is measured again before samples are analyzed and at the beginning of each subsequent 12 hour shift. If the breakdown check fails, then injection port/column maintenance is required

10.2.7. Client samples and QC samples may be analyzed after the analysis of the performance check. Sample analysis may proceed for up to 20 samples or 12 hours prior to analysis of another calibration verification (whichever is more frequent).

10.2.8. A Continuing Calibration Verification (CCV) for the 21 single response pesticides plus surrogates must be analyzed and evaluated every 12 hours or 20 samples (whichever is more frequent) as described in Section 9.2.4.4.. This is accomplished by running the midpoint calibration standard as a CCV (Pest Mix 100 ppb check standard and Mirex 100 ppb check standard; see Section 7.2.2.1). The calculated concentration for each compound in the CCV must be +/- 20 % of the expected concentration. Any samples analyzed after a failing CCV must be reanalyzed under a passing CCV. If, after performing instrument maintenance, the reanalysis of a CCV fails criteria, a new initial calibration range must be analyzed (see Section 9.2.4.2). Any data reported against a failing CCV must have a Non-Conformance Memo detailing the issue.

10.2.9. Analytical Sequence: The automation of GC runs is accomplished via the "SEQUENCE" macro of the Chemstation. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the check standards, evaluate the instrument status, and then complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro.

Example Analytical Sequence	
1. Hexane	12. Mirex 1 (10 ppb) (if needed)
2. Instrument Blank	13. Mirex 2 (20 ppb) (if needed)
3. Endrin/DDT Breakdown	14. Mirex 3 (100 ppb) (if needed)
4. Pesticide Mix 1 (2.5 ppb)	15. Mirex 4 (250 ppb) (if needed)
5. Pesticide Mix 2 (50 ppb)	16. Mirex 5 (500 ppb) (if needed)

Example Analytical Sequence	
6. Pesticide Mix 3 (100 ppb) 7. Pesticide Mix 4 (250 ppb) 8. Pesticide Mix 5 (500 ppb) 9. Chlordane (1000 ppb) 10. Toxaphene (1000 ppb) 11. Pesticide ICV (100 ppb)	17. Mirex ICV (100 ppb) (if needed) 18. Instrument Blank 19. Endrin/DDT Breakdown 20. Pest Mix 3 (100 ppb)…CCV 21. Mirex 3 (100 ppb) …CCV (if needed) 22. 20 or fewer samples or 12 hours 23. Instrument Blank 24. Endrin/DDT Breakdown 25. Pest Mix 3 (100 ppb)…CCV 26. Mirex 3 (100 ppb) …CCV (if needed) 27.20 or fewer samples or 12 hours

10.3. Dual Column Approach

10.3.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. If the difference between the dual columns results in ≤40% RPD report the higher concentration.

10.3.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows. Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period.

10.3.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P*. **Exception:** NJDEP DKQP protocols require reporting the higher concentration in all instances.

10.3.4. If the surrogates on one column are >40% RPD compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.3.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.3.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.3.6.1. An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on

both columns. In this case the non-detect results may be reported from the compliant column.

10.3.7. In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.

10.3.8. In summary, the flow chart in Attachment 1 presents a recommended rational approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.4. Extract Cleanups

10.4.1. Cleanup methods are dictated by the original sample matrix and the parameters being determined.

10.4.2. Cleanup of all water samples, if needed, is performed using Florisil (Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision) and TBA sulfite (Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.3. Cleanup of all soil samples is conducted using Florisil (Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision) and, if needed, TBA sulfite (Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.4. Check Florisil prior to use to assure quantitative recovery of target analytes. (see Section 4.3.2 above and Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B,

10.5. Data Processing and Documentation

10.5.1. Before the analysis sequence is initiated the GC Maintenance logbook must be filled out. It should contain the following information: date, injector temp,

oven temp, detector temp, injector flow, signal A, signal B, analysts initials, and notes for any necessary repairs.

10.5.2. After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor, method, job number, QA number, extraction date, lab prep batch, Chrom batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

10.5.3. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and %moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).

10.5.4. Any compound concentration that exceeds the concentration of the calibration range must be diluted and re-analyzed.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculation of Sample Amounts (Internal Standard Procedure)

11.3.1.1 Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.1.2 Solid Samples

$$\text{Concentration (µg/kg)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of target analyte peak
Ais = Area of internal standard peak
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD):

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level i

11.7. Percent Difference (% D):

$$\%\ D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.8. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.9. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted.

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification:

The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution

prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage.

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710

Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method SW8000C: Determinative Chromatographic Separations,”, Test Methods for Evaluating Solid Wastes, SW846, Revision 3, March 2003.

15.2. United States Environmental Protection Agency, “Method 8081B, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, February 2007.

15.3. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. Eurofins Edison SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C,* most current revision.

15.5. Eurofins Edison SOP No. ED-ORP-018*: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B,* most current revision.

15.6. Eurofins Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

15.7. Eurofins Edison SOP No. ED-ORP-019*: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A,* most current revision.

15.8. Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision.

15.9. Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision.

15.10. Eurofins Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.11. Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*), most current revision.

15.12. Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), most current revision.

15.13. Eurofins Document CA-Q-W-008, ‘Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version.

15.14. Eurofins Corporate Quality SOP No. CA-Q-P-004, “Reporting Results for Methods that Require Second-Column Confirmation”, most current revision.

15.15. Eurofins Corporate Work Instruction No. CA-T-WI-008, “Minimum Requirements for Pesticide Analysis by SW846 Method 8081B”, most current revision.

15.16. Eurofins Corporate Policy CA-T-P-004, "Policy for Determining RT Windows for GC/ECD Tests", most current revision.

15.17. Eurofins Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005", most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Dual Column Reporting Flowchart

18.0. Revision History

Revision 7, dated 10/172022:

- Updated to Eurofins branding throughout document.
- Revised Section 8 to include requirement to treat samples from chlorinated sources with sodium thiosulfate
- Section 9.2.4.3: Included details of evaluation of initial calibration for Relative Standard Error (RSE).
- Section 11.6: added formula for calculation of RSE.
- Section 12.2 added: details the annual LLOQ verification requirement.

Revision 6, dated 03/25/2021:

- Added Section 10.5.3 which details how the reporting limit is established.
- Added Section 10.5.4 which discusses requirement to dilute analytes into range of calibration.

Revision 5, dated 10/12/2020:

- Edited throughout to reflect Eurofins branding and policy.
- Updated Lab Quality Manual section references as needed.

Revision 4, dated 09/13/2016:

- Section 9.2.4.1: completely rewrote this section to reflect requirements of Eurofins Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010
- Section 15 (References): added references to TestAmerica Corporate Technical Services Policy No. CA-T-P-005, "Policy for Determining RT Windows for GC/ECD

Tests" and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005".
- Attachment 1: RT Windows for Single Analytes/Surrogates: DELETED and renamed subsequent attachments.

Revision 3, dated 01/05/2016:

- Section 2.2: expanded to include summary of internal standard calibration.
- Section 7.2.2: added 1-Bromo-2-nitrobenzene (internal standard) to the list of standards (Restek 32279). Components and concentration added to table.
- Added Section 7.2.2.4 which describes the preparation of the internal standard solution. All subsequent sections renumbered accordingly.
- Section 7.2.2.1.1: added footnote to ICAL standard prep table detailing requirement to spike each standard with internal standard solution.
- Section 7.2.2.4: describe internal standard spiking protocol (20 ul of 5.0 ug/ml solution into all standards, QC extracts and field sample extracts.
- Section 9.1: added internal standard to 'Sample QC' table.
- Section 9.1.4: added following minimum requirements for surrogate evaluation and reporting: a) Both surrogates must have reportable results that meet the acceptance criteria; b)reported surrogates must be from a column with a passing CCV; c)At least one surrogate must pass on any column from which target analytes are identified and reported. (per corp "Minimum Requirements" document).
- Section 9.1.5: added this section which describes acceptance criteria for internal standards (retention time and response).
- Section 9.2.3: Added the following sentence: "'Samples' here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… ". Deleted the phrase 'and at the end of every sequence'. (both changes reflect 'Minimum Requirements for Pesticide Analysis' document).
- Added Section 9.2.4.1.3: "Updating absolute retention times: Update retention times with the retention times found in the opening CCV for that 12 hour period."
- Section 9.2.4.2: replaced 'external standard' with 'internal standard'. Added formula for calculation of response factors by internal standard method. Deleted text regarding calibration acceptance criteria from this section as it is duplicated in subsequent sections.
- Section 9.2.4.2.2: added phrase 'for any given compound.'
- Added section 9.2.4.2.5: describes resolution check.
- Sections 9.2.4.3 and 9.2.4.4: added statement concerning 80% resolution requirement.
- Section 9.2.4.4: changed 'ICV' to 'CCV' in last sentence.
- Section 10.1.2.13: Revised first sentence to read: ": HP Chemstation software is used for automation of runs and data acquisition."
- Section 10.2.2: corrected reference to Section 9.2.2.1 (was incorrectly listed as 7.2.2.1).
- Section 10.2.8: revised to clarify that CCV bracketing is not required and that only samples analyzed after a failing CCV must be reanalyzed (i.e., samples analyzed immediately prior to a failing CCV need not be reanalyzed).
- Section 10.3.2: added the following regarding updating retention times: "Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period."

- Section 11: added formulas for calculation of sample concentrations as well as %RSD, %D, % Recovery, RRF and dry weight correction.
- Section 15: removed reference to SOP No. ED-ORP-016*: Automated Soxhlet Extraction of Solid Samples – Pesticides/PCBs, SW846 Method 3541* as it is no longer is use.
- Section 15: added reference to TestAmerica Corporate Quality SOP No. CA-Q-P-004, "Reporting Results for Methods that Require Second-Column Confirmation"
- Section 15: added reference to TestAmerica Corporate Work Instruction No. CA-T-WI-008, "Minimum Requirements for Pesticide Analysis by SW846 Method 8081B".
- Section 15: added reference to TestAmerica Corporate Policy Memorandum No. CA-Q-QM-006, "Technical Guidelines for Analysis of Complex GC/ECD Chromatograms".

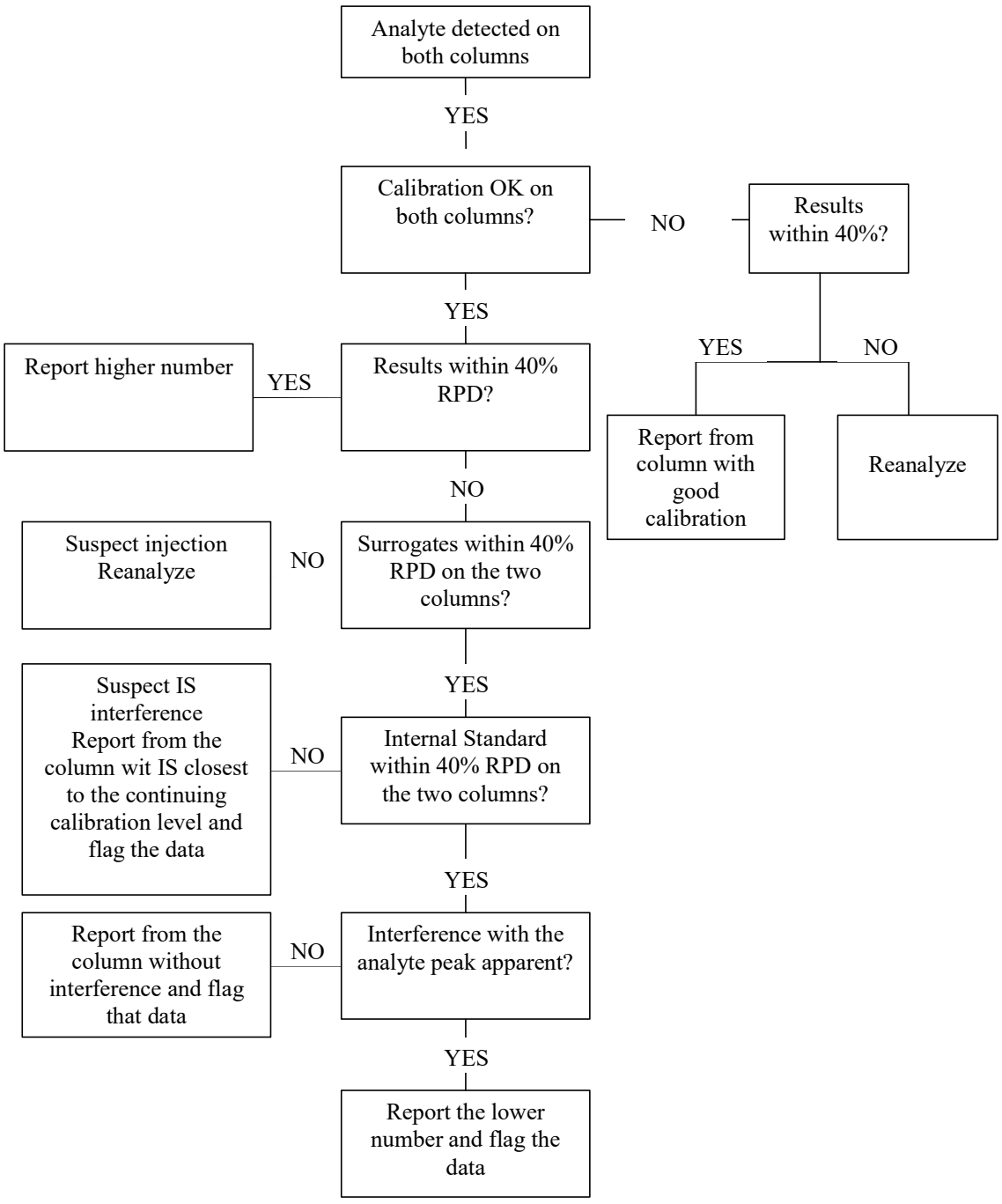
- Revision 2, dated 13 May 2015:
  - Section 1.1: updated RLs to reflect current lab practice.
  - Section 2.1 and throughout: revised initial aqueous/TCLP sample volumes to reflect current lab practice
  - Section 2.1.1: removed note describing option for Reduced Volume Extraction (RVE) as this is now standard lab practice.
  - Section 2.1.2 and throughout document: removed reference to prep by SW3541 as this method is no longer in use at Edison lab.
  - Section 4.3.2 and throughout: updated source of standards used for Florisil check.
  - Section 6.1.2.3.1: updated name of analytical GC columns currently in use.
  - Section 6.1.2.4.1: updated make-up gas to P-5 & Nitrogen.
  - Section 7.1.1: updated carrier gas to Hydrogen and make-up gas to P-5&Nitrogen.
  - Section 7.2.2 and throughout document: updated source and catalog numbers for analytical standards (Restek is now primary source of standards).
  - Section 7.2.2.1: updated standards prep instructions. Removed outdated references to standards and spiking mixes. Removed notes pertaining to RVE since this procedure is now standard and incorporated into instructions.
  - Section 7.2.2.10: added instructions for preparation of ICVs.
  - Section 8.1: adjusted water sample container from 1000ml to 250ml.
  - Section 9.2.2: removed references and instructions pertaining to the Pesticide Resolution Check (a CLP requirement we no longer perform).
  - Section 9.2.3.1: Added extensive discussion regarding the analysis of replicated CCVs.
  - Section 9.2.4.4: updated table to include new concentration for low standard (2.5 ppb).
  - Section 10.1.2.14 and throughout document: replaced references to Target with TestAmerica Chrom.
  - Section 10.2: made extensive revisions to discussion of the analytical sequence (removing duplicate entries and adjusting to reflect current procedures).
  - Section 10.3: added note concerning NJ DKQP requirements for reporting highest concentration of dual columns.
  - Section 15 (References): updated to include "Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version."
  - Added Sections 7.2.2.11 and 7.2.2.12 reflecting the addition and preparation of Internal standard

- Revision 1, dated 09 October 2012
  - Throughout: Revised LQM section number references to reflect the most current LQM revision.
  - Section 2.1.1: added description of reduced initial volume (125ml)/final volume (1ml) option.
  - Section 2.1.2: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546.*
  - Section 7.2.2.1.1 added preparation of 5ppb standard for the reduced volume method.
  - Section 9.2.2.2 Added 5ppb standard for the reduced volume method
  - Section 9.2.4.4 Added 5 ppb standard to the initial calibration for the reduced volume method
  - Section 10.2.6 Added 5 ppb to the Analytical sequence for the low level method Section 15.0: Removed reference to TestAmerica Edison SOP EDS-GEN-019, *Organic Calculations,* most current revision.
  - Section 15.0: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

- Revision 0, dated 02/17/2011: New

ATTACHMENT 1

Dual Column Reporting Flowchart
(Note: NJDEP DKQP requires reporting the higher concentration in all instances)



Title: SW846 Method 8082A, Analysis of Polychlorinated Biphenyls by Gas Chromatography

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Approvals (Signature/Date):

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1.0 Scope and Application

This method is used to quantify specific polychlorinated biphenyls (PCBs) as Aroclors (see Table 1 below) in extracts from aqueous, soil, sludge, leachate, wipe or oil matrices by direct injection dual capillary column gas chromatography using SW846 Method 8082A. An electron capture detector (ECD) is employed for detection.

1.1 Analytes, Matrix(s), and Reporting Limits

The specific analytes determined by this method are identified in Table 1.

Table 1
Polychlorinated Biphenyls

Compound Name	CAS Registry No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

The routine Eurofins Edison reporting limits (RLs) by analyte and matrix are summarized in Table 2 (below).

Table 2
Reporting Limits by Matrix

Parameter	Soil	Soil	Water	Leachate	Oil	Wipe
	Reporting Limits (ug/kg) LOW Level	Reporting Limits (ug/kg) MED Level	Reporting Limits (ug/L)	Reporting Limits (mg/L)	Reporting Limits (ug/kg)	Reporting Limits (ug/wipe)
Aroclor-1016	67	500	0.50	0.0050	1000	0.40
Aroclor-1221	67	500	0.50	0.0050	1000	0.40
Aroclor-1232	67	500	0.50	0.0050	1000	0.40
Aroclor-1242	67	500	0.50	0.0050	1000	0.40
Aroclor-1248	67	500	0.50	0.0050	1000	0.40
Aroclor-1254	67	500	0.50	0.0050	1000	0.40
Aroclor-1260	67	500	0.50	0.0050	1000	0.40
Aroclor-1262	67	500	0.50	0.0050	1000	------
Aroclor-1268	67	500	0.50	0.0050	1000	------

The most current MDLs and RLs for this method can be found in the active Eurofins LIMS (TALS) SW846 8082A Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Samples undergo a preparation step prior to analysis by SW846 Method 8082A. A measured volume or weight of sample (15 g for soil, 1 g for oil, 250 ml for water and TCLP/SPLP/ASTM leachates) is extracted using the appropriate matrix-specific sample extraction technique (reference the applicable Organic Sample Prep SOPs listed below). The extract is exchanged into hexane and concentrated to a final volume between 1 and 20 ml depending upon the prep technique used.

2.1.1. Aqueous and leachate samples are extracted at a neutral pH using SW846 Method 3510C (SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C).*

2.1.2. Wipe samples are extracted using SW846 Method 3550B: Sonication (*SOP No. ED-ORP-018: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B*)

2.1.3. Solid samples are extracted using SW846 Method 3546 (*SOP No. ED-ORP-0044: Procedure for the Microwave Extraction of Solids,SW846 Method 3546*).

2.1.4. Organic liquids are prepared using SW846 Method 3580A (*SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A*).

2.1.5. Extract cleanup steps are employed as need depending on the nature of the matrix interferences encountered. Suggested cleanups include SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*), SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*) and SW846 Method 3665A (*SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*) for heavy organic interferences.

2.2. After cleanup, the extract is analyzed by injecting a known volume of sample into a gas chromatograph equipped with a dual wide-bore fused silica capillary columns and dual electron capture detectors (GC/ECD). The GC is temperature programmed to separate and detect the analytes recovered during the extraction step. Quantitation is accomplished by comparing the area response of each target analyte relative to an internal standard established through a five-point initial calibration (six points for second order regression). Specific calibration and quality control steps are detailed in this SOP and meet the specification of SW846 Method 8082A.

2.3. Samples are analyzed only after all the necessary calibration and QC checks have been performed.

2.4. Acquired data from sample analysis is manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences from phthalate esters introduced during sample preparation can pose major difficulties for PCB determinations.

4.1.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting PCBs. Sulfur contamination should be expected with sediment samples. Employ SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*) for removal of sulfur.

4.3. Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*),

4.4. Interferences from other organic compounds can effectively be removed using a sulfuric acid treatment, SW846 Method 3665A (*SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*). This destructive technique can be employed only when the sample extract is being analyzed solely for PCBs (i.e., it is not to be used prior to analysis for pesticides).

4.5. Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, and DDD) will cause interference. A standard of the DDT analogs should be injected to determine which of the PCB or Aroclor peaks may be subject to interferences on the analytical columns used. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples. A System Performance Check (DDT/Endrin) is analyzed daily to assist in evaluation of potential DDT interference with this Aroclor 1254 peak.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas Chromatograph:

6.1.1 Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC), equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides and PCB's. All operations are as automated as possible with the equipment utilized.

6.1.2 Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles a single sample between the sample tray and the injector turret.

6.1.2.1 The sample is injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.

6.1.3 Liners: The injection port is each fitted with a replaceable, heavy-walled glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.

6.1.4 Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35°C and 350°C.

6.1.4.1 Two dissimilar columns are used for analysis. A Restek StxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column (or equivalent) is used for sample quantitation. The secondary column is a Restek StxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column (or equivalent).

6.1.5 Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.

6.1.5.1 Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas is fed from a supply other than the injection port.

6.2 Data System:

6.2.1 The data systems consist of Agilent Technologies GC Chemstation Revision A.08.02 and Agilent Technologies Enviroquant Chemstation G1701AA Version A.03.00 upgraded to A.03.02 which is used for acquisition and Eurofins Chrom (chromatography data processing software).

7. Reagents and Standards

7.1. Reagents

7.1.1. Gases: Ultra high purity (99.999%) Hydrogen is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. Ultra high purity (99.999%) Nitrogen is used as make-up gas. (Alternatively, ultra-high purity Helium with P-5 make-up gas may be used). Make-up gas is introduced to the GC via the make-up gas adapter at the end of the capillary column. Gases are supplied at tank pressures of 2000-2400 psig for a 300 cft tank. The tank pressure is regulated to an outlet pressure of 70 psig. Each tank is used until the tank pressure drops to less than 500 psig.

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the G.C. The traps are as follows:

- Hydrocarbon trap
- H_2O (moisture) trap
- O_2 scrubber

7.1.1.2. Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).

7.1.2. Solvents used in the extraction, clean up procedures and dilutions include Hexane, Methylene Chloride, and Acetone that are exchanged to Hexane prior to analysis. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis.

7.2. Standards

7.2.1. Standards are purchased as concentrated solutions. Standard compounds or mixtures for this analysis include an Aroclor 1016/1260 mix, Aroclor 1221, 1232, 1242, 1248, 1254, 1262, 1268 and the surrogate compound Decachlorobiphenyl (DCB) (packaged with the Tetrachloro-m-xylene (TCMX), a surrogate used in pesticide analysis).

NOTE: Two independent sources are used for quantitation standards and spiking standards

7.2.1.1. Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent.

7.2.2. Standards mixes and sources: *

Standard Name	Source	Concentration
TCMX/DCB Surrogate Calibration Mix	Restek 32000	200 ug/ml
TCMX/DCB Surrogate Spike Mix	Supelco 861275	10 ug/ml
Aroclor 1016 Calibration Standard	Supelco 48097	1000 ug/ml
Aroclor 1221 Calibration Standard	Restek 32007	1000 ug/ml
Aroclor 1232 Calibration Standard	Restek 32008	1000 ug/ml
Aroclor 1242 Calibration Standard	Restek 32009	1000 ug/ml
Aroclor 1248 Calibration Standard	Restek 32010	1000 ug/ml
Aroclor 1254 Calibration Standard	Restek 32011	1000 ug/ml
Aroclor 1260 Calibration Standard		1000 ug/ml
Aroclor 1262 Calibration Standard	Restek 32409	1000 ug/ml
Aroclor 1268 Calibration Standard	Restek 32410	1000 ug/ml
Aroclor 1660 Mix (Arolcors 1016 & 1260)	Restek 32039	1000 ug/ml
Aroclor 1016/1260 Calibration Standard (Second Source)	Restek 32039.sec	1000 ug/ml
1-Bromo-2-nitrobenzene (internal standard)	Restek 32279	1000 ug/ml

*Suppliers with equivalent standards may be used.

7.2.3. Aroclor 1016/1260 & Surrogate Calibration Standard Solution Preparation

Five levels of calibration standards are prepared using the above referenced Aroclor 1660 Mix (Restek – 23039) and TCMX/DCB Surrogate Calibration standard mix (Restek 32000). They are prepared as follows:

Final Concentration of Aroclor 1016/1260 (Concentration of DCB)	Volume (ul) of Aroclor 1660 Mix (1000 ug/ml)	Volume (ul) of TCMX/DCB Surrogate Calibration Mix (200 ug/ml)	Final Volume in hexane (ml)
50 ppb (25 ppb DCB) [1]	5	6.25	100
500 ppb (50 ppb DCB)	50	25	100
1000 ppb (100 ppb DCB)	1000	500	1000
1500 ppb (150 ppb DCB)	150	75	100

Final Concentration of Aroclor 1016/1260 (Concentration of DCB)	Volume (ul) of Aroclor 1660 Mix (1000 ug/ml)	Volume (ul) of TCMX/DCB Surrogate Calibration Mix (200 ug/ml)	Final Volume in hexane (ml)
2500 ppb (200 ppb DCB)	250	100	100

(1): The low level Aroclor 1016/1260 standard is 50 ppb and 12.5 ppb for the surrogate DCB (prepare by making a 2x dilution of the 100 ppb standard in hexane).

7.2.4. Surrogate Spiking Solution (soil and water)

A TCMX/DCB Surrogate Spike Mix is prepared by diluting 10 ml of Restek 32000 (see Table 1 above) to 200 ml of Acetone. Final solution concentration is 10 ug/ml. For reduced volume LVI preps a secondary dilution of this mix is utilized. This is prepared by diluting 20 ml of the 10 ug/ml solution to 100 ml acetone with a final solution concentration of 2 ug/ml.

7.2.5. Aroclor 1016/1260 Spiking Solution (soil, water and wipe)

An Aroclor 1660 Mix is prepared by diluting 10 ml of Restek 32039 (See Tables 1 and 2 above) to 100 ml acetone. Final solution concentration is 100 ug/ml. For reduced volume LVI preps a secondary dilution of this mix is utilized. This is prepared by diluting 20 ml of the 100 ug/ml solution to 200 ml with a final solution concentration of 20 ug/ml. used spiking soils and waters as received from Supelco without further dilution.

7.2.6. Individual Aroclor Calibration Solutions (1221, 1232, 1242, 1248, 1254, 1262 & 1268)

A 1000 ppb calibration standard is prepared for each remaining Aroclor from the stock standards detailed in Section 7.2.1. 200ul of 1000 ug/ml individual Aroclor solution and 100ul of 200 ug/ml TCMX/DCB is diluted to 200ml with Acetone. The final concentration of surrogates is 100 ppb.

7.2.7. Aroclor 1016/1260 Initial Calibration Verification (ICV) Standard Solution Preparation

A mid-point Aroclor 1016/1260 ICV standard is prepared using the second source Aroclor 1016/1260 Calibration Standard (Restek 32039.sec) detailed in Section 7.2.2. 1000 ul of 1000 ug/ml standard along with 500 ul of 200 ug/ml TCMX/DCB surrogate standard (Restek 32000) is diluted to 1000 ml with acetone for a final ICV concentration of 1 ug/ml (1000 ppb).

7.2.8. PCB Internal Standard Spike Mix (1 ug/ml)

The PCB 1 ug/ml internal standard spike mix is prepared by dilution 500ul of 1000 ug/ml of the 1-Bromo-2-Nitrobenzene standard (Restek 32279) in to 500 ml of Hexane. 20 ul of this solution is added to all standards, QC samples and field sample extracts prior to analysis.

7.2.9. System Performance Solution (3,3-DDT and Endrin at 0.25 ug/ml):

The DDT/Endrin performance solution is prepared by taking 250 ul of 500 ug/ml DDT/Endrin Mix (Supelco Catalog No 48282) and bringing it up to a volume of 500 ml with hexane.

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 250 ml	250 ml	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 ± 2°C	14 days to extraction; Analyze within 40 days of extraction	SW846

.

8.1. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

8.2. Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standard	every sample[3]	Response within -50% to +100% of most recent cal standard.

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method Blanks** are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. **Laboratory Control Sample (LCS):** A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to assess method performance and serves to determine whether the methodology is in control at the time of preparation and analysis. The recoveries of the LCS must fall within lab generated acceptance criteria. If the LCS recovery results are outside QC limits, the extract is reanalyzed. If upon reanalysis the recoveries remain outside of recovery limits the following evaluations are made:

- If LCS results fall outside the laboratory generated limits with low recoveries (refer to the current TALS Method Limit Group database), the LCS and all associated samples should be re-extracted and re-analyzed.

- If LCS results fall outside the laboratory generated limits with high recoveries (refer to the current TALS Method Limit Group database), the LCS and associated sample results may be reported with an Non-Conformance Memo (NCM) detailing the issue.

9.1.3. **Matrix Spike/Matrix Spike Duplicate (MS/MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to the current TALS Method Limit Group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated. If the LCS recoveries meet criteria the data is reported and a Non-Conformance Memo (NCM) is written.

9.1.4. **Surrogate Standards:** All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the DCB surrogate standard is calculated and compared to lab generated limits (refer to the current TALS Method Limit Group database). (Note: the surrogate must pass CCV criteria to be reportable and must be reported from the same

column as sample target analyte results). If the DCB recovery is outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.1.5. Internal Standard: The internal standard (1-bromo-2-nitrobenzne) must elute within 30 seconds of and have an area response of 50 to 100% as compared to the most recent preceding calibration standard.

9.2. Instrument QC

9.2.1. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.1.1. *Initial Calibration Range*: Aroclors 1016/1260 and the surrogate (DCB) are calibrated using a five-point calibration range using a minimum of five (5) peaks per Aroclor. The reporting limit (RL) is equal to the low point of the calibration range. The initial calibration block must include at least one level with Aroclor 1016 analyzed separately for pattern recognition purposes (note: this run does not need to be part of the actual calibration). If the 1016/1260 calibration meets the required average RF criteria all other Aroclors are then calibrated at the anticipated midpoint of the calibration range with a single point calibration using a minimum of five (5) peaks for each Arolcor (minimum of 3 peaks for Aroclor 1221). All peaks selected must be at least 25% the peak height of the largest peak in the Aroclor (except for Aroclor 1268 where the requirement is 10%). The following Aroclors can be analyzed together: 1016/1260, 1221/1254, 1232/1262 and 1242/1268. Standards are prepared following the instructions in Section 7.2.

9.2.1.2. *Initial Calibration Verification (ICV):* An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.7 and must be from a source separate from the standards used in the Initial Calibration Range.

9.2.2. Continuing Calibration Verification (CCV): A mid-point Continuing Calibration Verification (CCV) standard (typically Aroclors 1016/1260) must be analyzed after every 20 samples at minimum. If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260. If an Aroclor other than Aroclors 1016/1260 is detected a CCV of the identified Arolcor must be analyzed within 12 hours of the sample. If that CCV does not meet criteria the sample must be reanalyzed under an acceptable CCV using the detected Arolcor. The response factors for the CCV must be within +/- 20 % of the initial calibration RF.

9.2.3. Calibration Acceptance Summary

9.2.3.1. Retention Time Windows: Retention time (RT) windows must be determined for all analytes.

9.2.3.1.1 Initial determination of RT windows.

9.2.4.1.1.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration of the first CCV in the daily sequence, whichever is most recent.

9.2.3.1. Initial Calibration Range. The internal standard calibration technique is employed for this method. The response factor (defined as the ratio of the area to the standard concentration) is calculated for each characteristic peak in the Aroclor 1016/1260 standard at each calibration concentration. The percent relative standard deviation (% RSD) of the response factors for each individual peak in the Aroclor 1016/1260 mix on each column is then determined (both columns must pass calibration criteria).

9.2.3.1.1. Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

RF = Response Factor

9.2.3.1.2. Linear Calibration: If the % RSD is less than 20% over its working range for at least five peaks in the Aroclor 1016/1260 mix, the linearity of the range is assumed for all Aroclors over the same analytical range. Each individual peak's response factor is used for quantitation of all the samples and verification standards. The average of the value calculated for each individual peak is used to report the concentration in the samples.

9.2.3.1.3. Linear Calibration Using Least Squares Regression: If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r squared value must be > 0.99 for the calibration to be acceptable

9.2.3.1.4. Relative Standard Error: The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation. The average Relative

Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (*r*) and Coefficient of Determination (r_2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on *r.* As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point

9.2.3.2. Initial Calibration Verification (ICV):

9.2.3.2.1. After the initial calibration range has been analyzed, an Initial Calibration Verification standard is analyzed on each column to verify the validity of the initial calibration range. The ICV standard must be from a standard lot independent of the standards used in the initial calibration range. The verification standard for PCBs is the mid-range Aroclor 1016/1260 standard at 1000ppb. (See Section 7.2.7 for details on the preparation of the ICV).

9.2.3.2.2. At least five characteristic peaks of each Aroclor 1016/1260 plus surrogates in the ICV must be checked to verify the Initial Calibration Verification. The calculated concentration of the ICV must be within ±20%D of the expected concentration. Should the %D exceed 20%, the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.

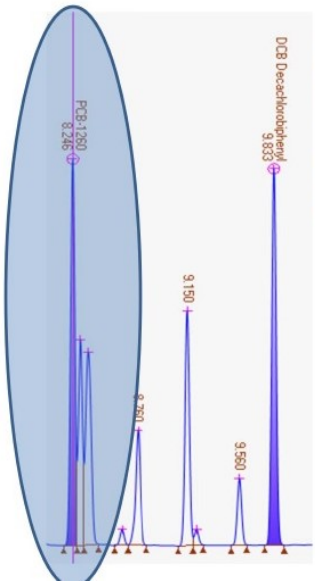
9.2.3.3. Continuing Calibration Verification (CCV) and System Performance Check:

9.2.3.3.1. On each day of analysis the System Performance Check (see Section 7.2.9) is analyzed to assist in the evaluation of possible DDT interferences with the last major peak in Aroclor 1254.

9.2.3.3.2. A mid-point Continuing Calibration Verification (CCV) standard (typically Aroclors 1016/1260) must be analyzed after every 20 samples at minimum. If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260.

9.2.3.3.3. If there are Aroclor hits in the associated samples then the calculated concentration of the CCV must be within ±20%D of the expected concentration on both columns. If there are no Aroclor hits in the associated samples one column may exceed the ±20%D criteria on the hight side (alternatively a low level Aroclor 1016/1260 standard may be analyzed to demonstrate adequate sensitivity. Should the foregoing criteria not be met the analyst must take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 20% after a single CCV reinjection, a new Initial Calibration Range must be analyzed. The surrogate (DCB) must also meet CCV criteria on both columns.

9.2.3.3.4. Resolution (Aroclor 1260): The CCV level Aroclor 1016/1260 standard must meet the following resolution critieria: Resolution (degree of overlap) for the triplet towards the end of the 1260 chromatogram must be <75% on <u>one</u> of the two columns used. The resolution requirement must be met for peak 1/2 and peak 2/3:



The circled triplet of peaks is observed towards the end of the 1260 pattern on columns such as CLP 1. Minimum resolution (degree of overlap) requirement between peak 1 / 2 and peak 2 / 3 is <75%. This chromatogram shows overlap of about 50% between peak 2 and 3, and 30% between peak 1 and 2.

Resolution (degree of overlap) is calculated as

[Height of the valley / (Sum of the two peak heights / 2)] x 100%

The acceptance criteria for the Initial Calibration Range, the ICV and the CCV are detailed in the table below.

Step	Standards	Type	Control Limit	Frequency
Method # 8082A				
Initial Calibration Range	50, 500, 1000, 1500 and 2500 ppb for Aroclor 1016/1260, 1000 ppb for all remaining Aroclors	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r^2 ≥0.990*	*Initially and as required when ICV or CCV do not meet requirements*
ICV	*1000 ppb*	*Average*	*± 20%D*	*Once after each initial calibration*
CCV	*1000 ppb*	*Average*	*± 20%D (see Section 9.2.3.4.2 for exceptions); Acceptable Resolution*	*Every 20 samples*
System Performance Standard			*For evaluation of DDT intereference with Ar1254*	*Once daily*

10. Procedure

10.1. Gas Chromatograph (GC) Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. General Instrument Operating Conditions:

10.1.2.1. **Injection System**: A splitless injection port with electronic pressure control (EPC) is used. Seventy-five seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

10.1.2.2. The EPC is used in the pressure Ramp mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	Rate	Final Pressure	Hold
25 psi	0.50 min	20psi/min	15 psi	2.00 min
		8 psi/min	12 psi	6.60 min
		10.0 min	16 psi	2.00 min

10.1.2.3. For PCB analysis the normal operating conditions of the injection port are as follows:

Injection Temperature:	250°C
Injection Port Pressure: Column flow:	25ml/min 33.2 ml/minute
Split vent flow:	60.0 ml/minute
Purge vent flow:	1.2 ml/minute
EPC:	Ramp pressure mode

10.1.2.4. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. This liner/glass wool combination provides many functions.

10.1.2.5. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.6. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.6.1. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.6.2. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all

column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.6.3. The septa should be changed each time the injection port is opened. Another routine maintenance operation to improve column performance is the removal of the first 3 cm of the column.

10.1.2.6.4. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.2.7. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.2.7.1. The oven program and pressure ramping for PCB analysis is employed for all columns as follows:

Initial Temp	Hold Time 1	Rate1	Temp1
164°C	0.0min	12°/min	234°C
Hold Time2	**Rate2**	**FinalTemp**	**FinalTime**
2.4 min	40°/min	325°C	1.5min

10.1.2.8. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.2.9. Chemstation: The Chemstation is utilized for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.2.10. Eurofins Chrom data processing software is used for the processing of the chromatography data files. Calibrations, verification standards and samples are processed and reviewed using this database. Chrom is integral to Eurofins LIMS (TALS) which is used to generate all reports.

10.2. Analytical Sequence

10.2.1. The instrument operating conditions should be set as detailed in Section 10.1.

10.2.2. Once instruments conditions have been established, the Initial Calibration Range, calibration verifications and retention time windows must be established Section 9.2.

10.2.3. The analytical sequence is established via the "SEQUENCE" macro of the Chemstation data system. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the calibration and/or calibration verification standards, evaluate the instrument status, and, finally, (if all meet criteria) complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro

10.2.4. An idealized analytical sequence including an Initial Calibration Range is presented in the table below.

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Idealized Analytical Sequence with Initial Calibration Range	
Injection Number	**Identification**
1	Hexane
2	Instrument Blank
3	Aroclor-1660 Level 1 Cal Std (50 ppb)
4	Aroclor-1660 Level 2 Cal Std (500 ppb)
5	Aroclor-1660 Level 3 Cal Std (1000ppb)
6	Aroclor-1660 Level 4 Cal Std (1500 ppb)
7	Aroclor-1660 Level 5 Cal Std (2500 ppb)
8	Aroclor-1221 Level 3 Cal Std (1000 ppb)
9	Aroclor-1232 Level 3 Cal Std (1000 ppb)
10	Aroclor-1242 Level 3 Cal Std (1000 ppb)
11	Aroclor-1248 Level 3 Cal Std (1000 ppb)
12	Aroclor-1254 Level 3 Cal Std (1000 ppb)
13	Aroclor-1262 Level 3 Cal Std (1000 ppb)
14	Aroclor-1268 Level 3 Cal Std (1000 ppb)
15	Initial Calibration Verification (Aroclor 1660)
16	Hexane
17	Continuing Calibration Verification (Aroclor1660)
18 thru 37	Client samples and QC Samples (MS/MSD, LCS, Method Blank)
19	Continuing Calibration Verification (Aroclor1660) (*every 20 samples*)

10.2.5. After each 20 samples a CCV standard mix must be analyzed. If this standard fails the criteria listed in Section 9.2.3.4, all samples analyzed during the previous period must be re-analyzed with a passing CCV.

10.2.6. PCB Data Reporting: The Chrom data system calculates the concentrations of the selected Aroclor Peaks. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and % moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).

10.2.7.1. The quantitative values for all confirmed analytes must agree within 40% between the primary column and the confirmation column.

10.2.7.2. If the quantitative values do not agree within 40%, the discrepancy must be noted in the report with a qualification

10.3 Dual Column Approach

NOTE: Data generated under the NJDEP DKQP requires the reporting of the higher concentration in all cases unless it can be demonstrated that interfering compounds are the cause of the higher results in which case the lower value can be reported with a narrative explanation. Other programs may also require a dual column reporting approach different than the one described below in which case the lab will report as required by that program.

10.3.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. Results are reported from the primary column unless the difference in concentration between the two columns results in ≥40% RPD in which case the lower concentration is reported (unless the client or program requirements dictate otherwise).

10.3.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows established from the most recent preceding calibration verification.

10.3.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P*.

10.3.4. If the surrogates on one column are very different (>40% RPD) compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.3.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the

compliant column. If the recovery falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.3.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.3.6.1. An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

10.3.7. In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.

10.3.8. In summary, the flow chart in Attachment 1 presents a recommended approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.4 Extract Cleanup

10.4.1. Cleanup methods are dictated by the original sample matrix and the parameters being determined.

10.4.2. Cleanup of all water samples, if needed, is performed using Sulfuric Acid Permanganate and/or TBA sulfite. Refer to Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision and Eurofins Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*, *SW846 Method 3665A,* most current revision. Blanks must also undergo cleanup following the same procedures as samples..

10.4.3. Cleanup of all soil samples is conducted using TBA sulfite and Sulfuric Acid Permanganate. Blanks must also undergo cleanup following the same procedures as samples.

10.4.4. Cleanup using Sulfuric Acid Permanganate effectively destroys the majority of organic material in the sample extract and should be used

only when PCB is the only analysis to be performed on the sample extract.

10.5 Documentation

10.5.1 Before the analysis sequence is initiated the GC Performance and Repairs logbook must be filled out. It should contain the following information: date, injector temp, oven temp, detector temp, column A flow, column B flow, signal A, signal B, analysts initials, and notes for any necessary repairs.

10.5.2 After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor method, job number, QA number, extraction date, lab prep batch, target batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculation of Sample Amounts (Internal Standard Procedure)

11.3.1 Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)

Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2 Solid Samples

$$\text{Concentration (µg/kg)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml

11.3.3 Wipe Samples

$$\text{Concentration (µg/kg)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(W)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
W	=	Wipe

The 1000 in the denominator represents the number of ul in 1 ml

11.4 Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of target analyte peak
Ais = Area of internal standard peak
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5 Percent Relative Standard Deviation (% RSD):

$$\%\ RSD = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6 Percent Difference (% D):

$$\overline{\%\ D} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7 Relative Standard Error (RSE)

$$\%\ RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level i

11.8 Percent Recovery (% R): Surrogates and Spikes

Concentration (or amount) found

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) added}}{\text{Concentration (or amount) added}} \times 100$$

11.9 Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted.

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL) The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification: The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.
  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This

material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

Teris Profile Number: 50016624
Onyx Profile WIP Number: 545240

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method SW8000C: Determinative Chromatographic Separations,", Test Methods for Evaluating Solid Wastes, SW846, Revision 3, March 2003.

15.2. United States Environmental Protection Agency, "Method 8082A, Organochlorine Pesticide by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 1, February 2007.

15.3. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. Eurofins Edison SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C,* most current revision.

15.5. Eurofins Edison SOP No. ED-ORP-018*: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B,* most current revision.

15.6. Eurofins Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

15.7. Eurofins Edison SOP No. ED-ORP-019*: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A,* most current revision.

15.8. Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision.

15.9. Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision.

15.10. Eurofins Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*, *SW846 Method 3665A,* most current revision.

15.11. Eurofins Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.12. Eurofins Corporate Work Instruction No. CA-T-WI-003, PCB Minimum Requirements, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Dual Column Approach

18.0. Revision History

Revision 7, dated 21 Oct 2022

- Section 4.5 added which details possible DDT and analogs interference with the last major DDT peak.
- Section 7.2.9: Added System Performance Standard (DDT/Endrin) for use in evaluating potential interference of DDT with last major Aroclor 1254 peak.
- Section 9.2.3.3.1: Added requirement to analyze System Performance standard daily for evaluation of DDT interference with Aroclor 1254 quantitation.

Revision 6 dated 17 Oct 2022

- Updated to Eurofins branding, removed TestAmerica references
- Added Section 8.2 to include requirement to treat samples from chlorinated sources with sodium thiosulfate
- Section 9.2.3.1.4: Section 9.2.4.3: Included details of evaluation of initial calibration for Relative Standard Error (RSE).
- Section 11.7: added formula for calculation of RSE.
- Section 12.2 added: details the annual LLOQ verification requirement.

Revision 5 dated 01 Apr 2020

- Updated to Eurofins branding
- Section 7.2.5: added wipe matrix
- Added Section 11.3.3 – calculation for wipe samples

Revision 4 dated 15 Dec 2016

- Section 7.2.8: clarified text to more accurately describe the PCB Internal Standard Mix.

Revision 3 dated 16 May 2016

- Section 2.1: removed SW846 3550 as a prep method for solids and added it as a prep method for wipe samples.
- Section 2.1.1 and 15.0: removed reference to SW846 Method 3520C (SOP No. ED-ORP-003: *Extraction of Semi-Volatile Organic Compounds in Water by Continuous Liquid-Liquid Extraction, SW846 Method 3520C).*
- Section 2.2: expanded to include reference to internal standard calibration.
- Section 7.1.1: revised to clarify that the primary carrier:make-up gas combo is hydrogen and nitrogen.
- Section 7.2.2: added 1-Bromo-2-nitrobenzene (internal standard) to the list of standards (Restek 32279). Components and concentration added to the table
- Added Section 7.2.8 which describes the preparation of the internal standard solution.
- Section 9.1: added internal standard to 'Sample QC' table
- Section 9.1.2: completely re-written to clarify LCS evaluation and corrective action.
- Section 9.1.4: clarified that surrogates must pass CCV criteria in order to be reportable and must be reported from the same column as target analyte results.
- Added Section 9.1.5 which describes acceptance criteria for internal standards (retention time and response)
- Section 9.2.1.1: added following sentence for clarity and compliance with TestAmerica PCB Minimum Requirements document (WI No. CA-T-WI-003): "All other Aroclors are calibrated using a single point calibration up to 8 peaks at the anticipated midpoint of the calibration range if the 1016/1260 calibration meets the required average RF criteria." Added language detailing which Aroclor standards can be analyzed together.
- Section 9.2.1.1: added this sentence: "The initial calibration block must include at least one level with 1016 analyzed separately for pattern recognition purposes (note: this run does not need to be part of the actual calibration)."
- Section 9.2.1.1: clarified that a minimum of five peaks are required for quantitation of each Aroclor (except for Aroclor 1221 which requires a minimum of three peaks). Added requirement that selected peaks must be at least 25% of height of largest peak in Aroclor (except Aroclor 1268 where the requirement is 10%).
- Section 9.2.2: Revised first sentence to read: "A mid-point Continuing Calibration Verification (CCV) standard must be analyzed after every 20 samples at minimum," (i.e., removed the closing CCV requirement and 12 hour requirement).
- Section 9.2.2: clarified that the CCV typically consists of a 1016/1260 standard. Also added the following sentence: "If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260."
- Section 9.2.2: added requirement to analyze a CCV for each detected Aroclor within 12 hours of detection.

- Table 2: updated with the expected RT acceptance criteria from Minimum Requirements document (0.03 minutes).
- Section 9.2.3.2: Revised 'External standard' to 'Internal Standard'. Emphasized that both columns must pass calibration criteria.
- Section 9.2.3.2.2: revised to require that 5 peaks must be selected (was 3) and evaluated for initial calibration (per corporate Minimum Requirements).
- Section 9.3.3.3.1: corrected section reference for preparation of ICV (was Section 7.2.9; now Section 7.2.7);
- Section 9.2.3.3.2: revised first sentence to read: "At least five characteristic peaks of each Aroclor 1016/1260 plus surrogates in the ICV must be checked to verify the Initial Calibration Verification." (it had previously required three peaks).
- Section 9.2.4.3.1: updated text reflect corporate Minimum Requirements document.
- Added Section 9.2.3.4.2: more fully describes CCV acceptance criteria and corrective actions. Updated table with calibration criteria.
- Added Section 9.2.3.4.3: details resolution requirements per the corporate Minimum Requirements document.
- Section 10.2.5: revised to remove 12 hour requirement (replaced solely with 20 sample requirement) and bracketing requirement. Corrected section reference for CCV acceptance criteria.
- Section 11: completely re-written to include internal standard result calculations and other required QC calculations.
- Section 15 (References): added following document reference: TestAmerica Corporate Work Instruction No. CA-T-WI-003, PCB Minimum Requirements, most current revision.
- Attachment 1: revised completely.

- Revision 2, dated 08 Jun 2015

  - Section 1.1: Corrected Method Limit Group (MLG) reference to 8082 (was incorrectly listed as 8081B,
  - Table 2: updated RLs for leachates from 0.0050 mg/L to 0.00050 mg/L.
  - Throughout document: updated the default initial volumes for aqueous preps (250 ml) and leachate (TCLP/SPLP/ASTM) preps (250 ml).
  - Throughout document: removed any notes referencing option for 'reduced volume' extractions since this is now standard as defined in SOP.
  - Section 2.2.2: replaced SW846 3541 (Soxtherm) prep with SW846 3546 (Microwave) prep.
  - Section 6.2.1 (and throughout document): replaced Target software references with references to TestAmerica's Chrom chromatography data processing software.
  - Section 7.2.2 and throughout document: updated source of standards from Supelco to Restek.
  - Section 7.2.3 and throughout document revised concentration of low calibration standard to 50 ppb.
  - Section 7.29: updated ICV prep instructions using Restek standards.
  - Section 8: updated sample container from 1000ml to 250ml; updated minimum sample size to 250ml.
  - Section 10.3: added note explaining NJDEP DKQP requirement to report the higher concentration in all cases.

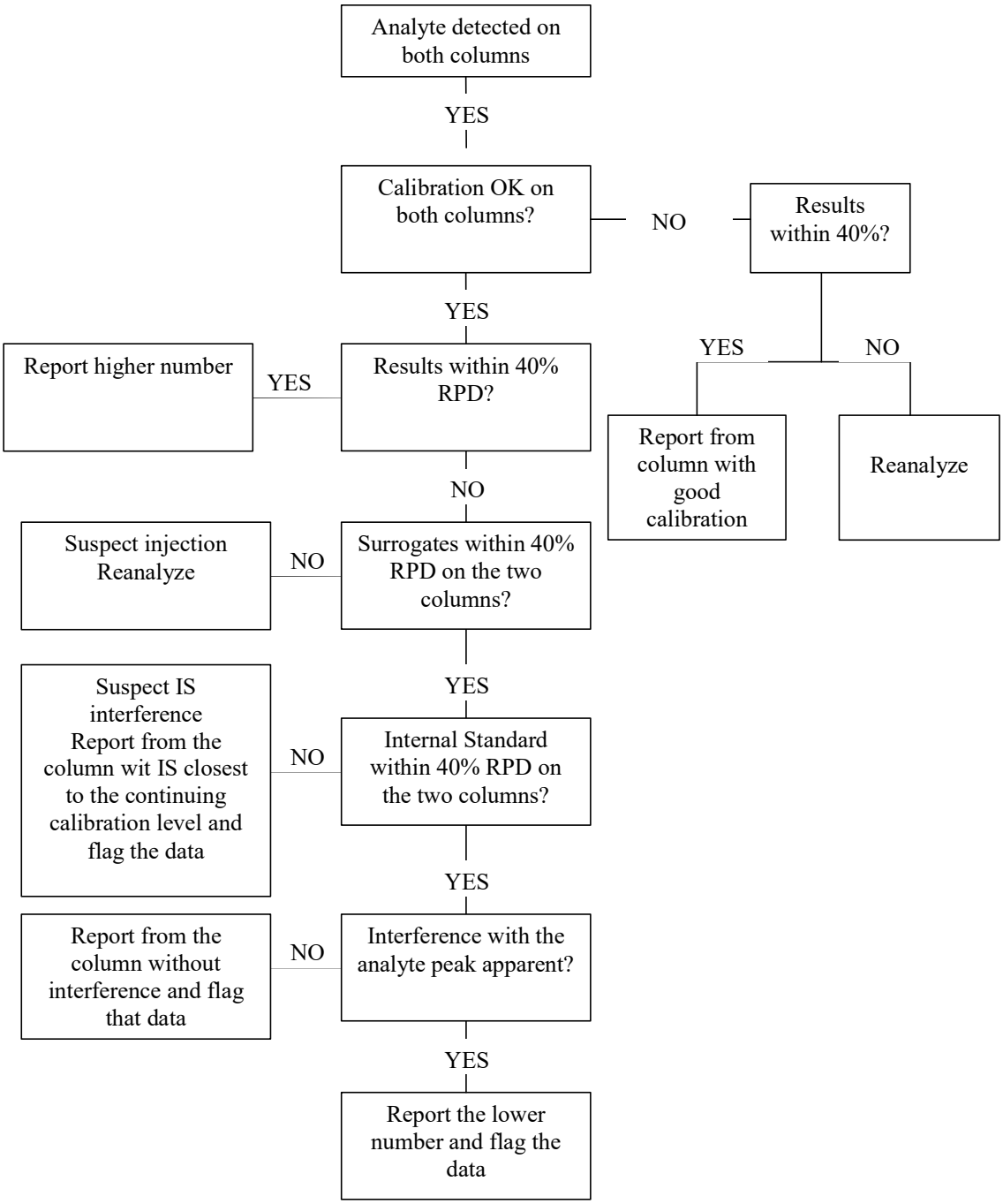
- Section 10.3 and Attachment 1: revised to reflect current TestAmerica dual column reporting rules.
- Section 15: removed outdated references.

- Revision1, dated 11 October 2012

  - Throughout document: Revised LQM section references to reflect the most current LQM revision
  - Revised Table 2 to reflect RLs for reduced initial/final volume prep option.
  - Section 2.1.1: added description of reduced initial volume (125ml)/final volume (1ml) extraction option.
  - Section 2.1.2 and Section 15.0: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546.*
  - Section 7.2.3: added procedure for prep and analysis of a lower level Aroclor 1016/1260 standard (50 ppb) and for the surrogate DCB (12.5 ppb) when analyzing samples prepped using the reduced initial/final volume procedure.
  - Section 9.2.3.4.1: Added reference to 50 ppb 1016/1260 ICAL standard for the reduced initial/final volume method.
  - Section 10.2.4: Added 50 ppb 1016/1260 ICAL standard to the sequence for the reduced initial/final volume method

- Revision 0, dated 02/16/2011: NEW

Attachment 1
Dual Column Approach


Analyte detected on both columns
YES
Calibration OK on both columns?
NO
Results within 40%?
YES
NO
YES
Report higher number
YES
Results within 40% RPD?
Report from column with good calibration
Reanalyze
NO
Suspect injection Reanalyze
NO
Surrogates within 40% RPD on the two columns?
YES
Suspect IS interference Report from the column wit IS closest to the continuing calibration level and flag the data
NO
Internal Standard within 40% RPD on the two columns?
YES
Report from the column without interference and flag that data
NO
Interference with the analyte peak apparent?
YES
Report the lower number and flag the data

Title: Analysis of Organochlorine Herbicides by SW846 Method 8151A

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes Eurofins Edison's procedure for the analysis of select organochlorine herbicides in solid and aqueous matrices by USEPA SW846 Method 8151A using dual capillary column gas chromatography (GC) with electron capture detector (ECD). Water samples are prepared using the extraction techniques detailed in Eurofins Edison SOP No. ED-ORP-015, *Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A,* current revision. Solid sample extraction procedures are detailed in Eurofins Edison SOP No. ED- ORP-023, *Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A,* current revision. Table 1 (below) lists the method target compounds as well as the analytical reporting limit for each compound by matrix.

Table 1: Analytes and Reporting Limits

Parameter	CAS No.	Soil Reporting Limits (ug/kg)	Water Reporting Limits (ug/L)	Leachate Reporting Limits (ug/L)	Waste Reporting Limits (ug/kg)
2,4-D	94-75-7	17	0.50	0.080	33
2,4,5-TP (Silvex)	93-72-1	17	0.50	0.080	33
2,4,5-T	93-76-5	17	0.50	0.080	33
Dalapon	75-99-0	17	0.50	n/a	33
Dinoseb	88-85-7	17	0.50	n/a	33
2,4-DB	94-82-6	17	0.50	n/a	33
DIcamba	1918-00-9	17	0.05	n/a	33
Dichlroprop	120-36-5	17	0.05	n/a	33
MCPA	94-74-6	1700	50.0	n/a	3300
MCPP	93-65-2	1700	50.0	n/a	3300
Pentachlorophenol	87-86-5	17	0.05	n/a	33
Picloram	1918-02-01	17	0.05	n/a	33

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 9 (Review of Work) and 19 (Test Methods and Method Validation) in the Eurofins Edison Quality Assurance Manual.

2.0 Summary of Method

2.1. A measured volume or weight of sample (30 g for soil, 1000 ml for water, 15 ml for leachates) is extracted using the appropriate preparation SOP (see Section 1.1). The final volume is 10 ml in hexane (soil and water) and 5 ml in hexane (leachates).

2.2. Water samples are extracted with diethyl ether and then esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.3. Soil and waste samples are extracted and esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.4. Samples are analyzed after all the necessary calibration and QC checks have been performed as described herein. Since quantitative values for organochlorine herbicide analyses are based on the methyl ester derivatives of the free acid herbicides, the results must be adjusted for the difference in molecular weight between the free acid and the methyl ester. (See Attachment 1 for Conversion Factors)

2.5. All data undergoes two documented levels of review prior to release to clients.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks.

4.1.1. Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by rinsing it with the last solvent used in it. This should be followed by detergent washing with hot water and rinses with tap water, then with organic-free reagent water. Glassware should be solvent-rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used. For complete details reference Eurofins Edison SOP No. ED-GEN-013, *Glassware Cleaning*, most current revision.

4.1.2. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

4.2. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably depending upon the nature and diversity of the material being analyzed.

4.3. Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure. The determination using pentafluorobenzylation is more sensitive, and more prone to interferences from the presence of organic acids or phenols than by methylation.

4.4. Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of Dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids. Strong organic acids react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

4.5. Organochlorine herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

4.6. Sample extracts should be dry prior to methylation or poor recoveries may result.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation:

6.1.1. The system used is an Agilent Technologies (Avondale, PA) model 5890A or 6890 Gas Chromatograph (GC). It is equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine herbicides. All operations are as automated as possible with the equipment utilized.

6.1.2. Injection system: Dual tower Agilent Technologies 7673 Autosampler, or equivalent. Sample injection is accomplished by dual autoinjectors. A robot arm that shuttles samples between the sample tray and the injector turret services the auto injectors.

6.1.3. The samples are injected into a split/splitless injection port. The injection port is normally operated in splitless mode during injection with electronic pressure control (EPC) providing for constant flow during oven temperature programming.

6.1.4. Liners: The injection ports are each fitted with replaceable, heavy-walled glass liners. Each liner contains a plug of Hexane extracted, Teflon wool approximately 1 cm in length. The Teflon wool is positioned in the liner 1 cm from the bottom for the 5890 system. The Teflon wool is positioned in the middle of the goose neck of the siltek liner used in the 6890 system.

6.1.5. GC Oven: capable of integrated temperature control between 35°C and 400°C. Temperature programming of the gas chromatograph is employed.

6.1.6. Two dissimilar columns are used for analysis. An Agilent Technologies DB-5, 30m x 0.53mm ID column is used for sample quantitation. The secondary column is an Agilent Technologies DB-608, 30m x 0.53mm ID megabore column.

6.1.7. Detectors: The GC is equipped with dual Electron Capture Detectors (ECD), one for each column. Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron

plasma. This is in addition to the gas exiting the column. The make-up gas consists of Ultra high purity (99.999%) Argon (95%)/Methane (5%) (a.k.a. P-5 Mixture) (P-5) and is fed from a supply separate from the carrier or injection port gas streams.

6.1.8. Data System:

6.1.8.1 **Chemstation:** The Chemstation is utilized for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

6.1.8.2 **Eurofins Chrom** chromatography data processing software package. Chrom is used for post acquisition GC data processing, reporting and storage and is fully integrated with Eurofins's LIMs (TALS).

6.1.9. Final processed data is uploaded to the Eurofins LIMS (TALS).

6.2. Supplies

6.2.1 4mm splitless sleeve (Restek Catalog # 11868-773 or equivalent) - This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

6.2.2 0.8mm ID, Gold Inlet Seal (Restek Catalog # 21241 or 21318 or equivalent)

6.2.3 Snoop Leak Detector solution or equivalent.

6.2.4 Gas-tight syringes (various sizes)

6.2.5 Injection port septa

6.2.6 Pre-silanized glass wool (Supelco 2-0411 or equivalent)

6.2.7 Syringes, Assorted sizes 10ul - 1000ul; gas-tight

6.2.8 Bottles, 10 and 5ml amber screw cap with Teflon liner

6.2.9 Vials, 2ml amber screw cap with Teflon liner

6.2.10 Wheaton microvials 100ul or equivalent

7.0 Reagents and Standards

7. 1. Reagents

7.1.1. **Gases:** Ultra high purity (99.999%) Helium is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. Ultra high purity (99.999%) Argon (95%) / Methane (5%) (a.k.a. P-5 Mixture) is used as make-up gas. It is introduced to the GC via the make-up gas adapter at the end of the capillary column. They are supplied by M-G Industries (Valley Forge, PA). Both gases are supplied at tank pressures of 2000-2400 psig., for a 300 cft. tank. The tank pressure is regulated to an outlet pressure of 70 psig. Each tank is used until the tank pressure drops to less than 500 psig.

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the GC The traps are as follows:

7.1.1.1.1. Hydrocarbon trap
7.1.1.1.2. H_2O trap
7.1.1.1.3. O_2 scrubber

7.1.2. All sample extracts are in hexane (reference the sample prep SOPs detailed in Section 2.0 above).

7.1.2.1. Hexane, Pesticide Grade, Baxter 217-4 or equivalent.

7.1.3. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7. 2. Standards

7.2.1. Standards are purchased as concentrated solutions (see Section 7.2.2). Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.3.

7.2.2. **Standard mixes and sources:**

Table 2:
Herbicide Standard Mixes and Sources*

Standard Name ("Lab Name")	Concentration (ug/ml)	Source
Acid Herbicide Spiking Mix 2 ("Spiking Mix")	varies (in acetone)	Supelco Catalog # 861259
Underivatized Chlorinated Herbicides ("10 Compound Spiking Mix")	Varies (in methanol)	Accustandard Catalog #M-8150A)
EPA 8270 Herbicide Ester Mix ("Calibration Mix")	2000 (in hexane)	Supelco Catalog # 48474
Pentachloroanisole ("Pentachloroanisole")	100 (in methanol)	Accustandard Catalog # P-199S
Picloram methyl ester ("Picloram methyl ester")	Neat	Chem Service Catalog # F2155
Methyl Derivatives of Chlorinated Herbicides ("Calibration Mix - 10 compound")	Varies (in methanol)	AccuStandard Catalog # M-8150
Pentachlorophenol ("Pentachlorophenol spike")	1000	Spex Certiprep Catalog #

Table 2: Herbicide Standard Mixes and Sources*		
Standard Name ("Lab Name")	**Concentration (ug/ml)**	**Source**
		S-2950
Picloram (4-amino-3,5,6-trichloropicolinic acid) ("Picloram Acid Spiking Standard")	Neat	Chem Service. Catalog No. F2041
2,4-DCAA Acid Spike Mix ("Surrogate Spiking Solution")	100 (in acetone)	Supelco Catalog # 861271
2,4-DCAA methyl ester ("Surrogate Solution")	1000 (in hexane)	Supelco Catalog # 861272

* May be substituted with equivalent standards from alternate sources.

Table 3: Components of Herbicide Standard Mixes			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
2,4-dichlorophenoxyacetic acid (2,4-D)	Supelco 861259	Spiking Mix	50
2-(2,4,5-Trichlorophenoxy)propionic acid (2,4,5-TP aka Silvex)	Supelco 861259	Spiking Mix	20
2,4,5-Trichlorophenoxyacetic acid (2,4,5-T)	Supelco 861259	Spiking Mix	20
2,4-D methyl ester	Supelco 48474	Calibration Mix	2000
2,4,5-T methyl ester	Supelco 48474	Calibration Mix	2000
Silvex methyl ester	Supelco 48474	Calibration Mix	2000
2,4-D methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dalapon methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
2,4-DB methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dicamba methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dichloroprop methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dinoseb methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
MCPA methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	10000
MCPP methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	10000
2,4,5-T methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
2,4,5-TP methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
2,4-D	Accustandard M-8150A	Spiking Mix – 10 compound	100
2,4-DB	Accustandard M-8150A	Spiking Mix – 10 compound	100
2,4,5-T	Accustandard M-8150A	Spiking Mix – 10 compound	100
Silvex	Accustandard M-8150A	Spiking Mix – 10 compound	100

Table 3:
Components of Herbicide Standard Mixes

Parameter	Catalog Nos.	Lab Standard Name	Concentration (ug/ml)
Dalapon	Accustandard M-8150A	Spiking Mix – 10 compound	100
Dicamba	Accustandard M-8150A	Spiking Mix – 10 compound	100
Dichloroprop	Accustandard M-8150A	Spiking Mix – 10 compound	100
Dinoseb	Accustandard M-8150A	Spiking Mix – 10 compound	100
MCPA acid	Accustandard M-8150A	Spiking Mix – 10 compound	10000
MCPP acid	Accustandard M-8150A	Spiking Mix – 10 compound	100000
Pentachloroanisole	Accustandard P-199S	Pentachloroanisole	100
Pentachlorophenol	Spex Certiprep S-2960	Pentachlorophenol spike	1000
Picloram methyl ester	Chem Service Catalog # F2155	Picloram methyl ester	neat
Picloram (4-amino-3,5,6-trichloropicolinic acid)	Chem Service. Catalog No. F2041	Picloram Acid Spiking Standard	neat
2,4-Dichlorophenylacetic Acid	Supelco 861271	Surrogate Spiking Solution	100
2,4-Dichlorophenylacetic Acid methyl ester	Supelco 861272	Surrogate Solution	1000

7. 3. Standards Preparation

7.3.1. All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware.

7.3.2. Total Herbicide Calibration Standards:

7.3.2.1 Total Herbicide Calibration Standards (10 compound analysis): The ten (10) compound/surrogate total herbicide calibration standard range is prepared when analyzing for the expanded list of herbicides. Table 4 details the preparation of the five calibration concentration levels using the 10 compound calibration mix. The standards are prepared using a methyl ester source with the final concentration adjusted to the free acid equivalent concentration using the conversion factors detailed in Attachment 1.

Table 4:
Preparation of Total Herbicide Calibration Standards (10 compound analysis)

Parameter	Level 1 Conc. (ug/ml) [1] 25.0 ul '8151-Herbicide Mix (10 compounds)' & 5ul 'Surrogate Solution" into 50ml Hexane	Level 2 Conc. (ug/ml) [1] 125 ul '8151-Herbicide Mix (10 compounds)' & 25ul 'Surrogate Solution" into 50ml Hexane	Level 3 Conc. (ug/ml) [1] 375 ul '8151-Herbicide Mix (10 compounds)' & 75ul 'Surrogate Solution" into 50ml Hexane	Level 4 Conc. (ug/ml) [1] 750 ul '8151-Herbicide Mix (10 compounds)' & 150ul 'Surrogate Solution" into 50ml Hexane	Level 5 Conc. (ug/ml) [1] 750 ul '8151-Herbicide Mix (10 compounds)' & 150ul 'Surrogate Solution" into 25ml Hexane
2,4-D methyl ester	0.047	0.235	0.705	1.41	2.82
2,4,5-T methyl ester	0.047	0.235	0.705	1.41	2.82
2,4,5-TP (Silvex) methyl ester	0.048	0.238	0.712	1.42	2.85
Dalapon methyl ester	0.046	0.228	0 .682	1.36	2.73
2,4-DB methyl ester	0.047	0.235	0.705	1.41	2.82
Dicamba methyl ester	0.047	0.235	0.705	1.41	2.82
Dichloroprop methyl ester	0.047	0.235	0.705	1.41	2.82
Dinoseb methyl ester	0.047	0.235	0.705	1.41	2.82
MCPA methyl ester	4.67	23.4	70.1	140	280
MCPP methyl ester	4.67	23.4	70.1	140	280
2,4-DCAA methyl ester (surr)	0.096	0.478	1.43	2.86	5.73

[1] The calculated final standard concentrations have been converted from the methyl ester concentration to the free acid equivalent concentration using the factors found in Attachment 1

7.3.2.2 Pentachlorophenol Calibration Standards: A pentachlorophenol /surrogate total herbicide calibration standard range is prepared when analysis is required for this compound. Table 5 details the preparation of the five calibration concentration levels. The standards are prepared using a source containing the derivatized form of the compound (pentachloroanisole) the final concentration adjusted to the free acid equivalent concentration using the conversion factors detailed in Attachment 1.

Table 5:
Preparation of Pentachlorophenol Calibration Standards

Parameter	Level 1 Conc. (ug/ml) [1] 50 ul 'Pentachloroanisole' & 10ul 'Surrogate Solution" into 100ml Hexane	Level 2 Conc. (ug/ml) [1] 25 ul 'Pentachloroanisole' & 5ul 'Surrogate Solution" into 10ml Hexane	Level 3 Conc. (ug/ml) [1] 75 ul 'Pentachloroanisole' & 15ul 'Surrogate Solution" into 10ml Hexane	Level 4 Conc. (ug/ml) [1] 150 ul 'Pentachloroanisole' & 30ul 'Surrogate Solution" into 10ml Hexane	Level 5 Conc. (ug/ml) [1] 300 ul 'Pentachloroanisole' & 60ul 'Surrogate Solution" into 10ml Hexane
Pentachlorophenol	0.048	0.237	0.712	1.42	2.85
2,4-DCAA methyl ester (surr)	0.096	0.478	1.43	2.87	5.73

[1] The calculated final standard concentrations have been converted from the deriviatized concentration to the free acid equivalent concentration using the factors found in Attachment 1

7.3.2.3 Picloram Methyl Ester Calibration Standards: A Picloram methyl ester/surrogate calibration standard range is prepared when analysis is required for this compound. Table 6 details the preparation of the five calibration concentration levels. The standards are prepared using a source containing the picloram methyl ester and the final concentration is adjusted to the free acid equivalent concentration using the conversion factors detailed in Attachment 1.

7.3.2.3.1 Prior to the preparation of the picloram calibration standards a working stock solution must be prepared using the neat material described in Table 2. This working stock solution is prepared at 9000 ug/ml by dissolving 9.00 grams of neat picloram methyl ester into 1000 ml of hexane using class A volumetric glassware. This solution is used as described in Table 6.

Table 6:
Preparation of Picloram Calibration Standards

Parameter	Level 1 Conc. (ug/ml) [1] 20 ml of Level 3 Picloram methyl ester into 100ml Hexane	Level 2 Conc. (ug/ml) [1] 33 ml of Level 3 Picloram Methyl ester into 100ml Hexane	Level 3 Conc. (ug/ml) [1] 8.5 ul Picloram Methyl ester stock (9000ug/ml) & 150 ul 'Surrogate Solution" into 100ml Hexane	Level 4 Conc. (ug/ml) [1] 17 ul Picloram Methyl ester stock (9000ug/ml) & 300 ul 'Surrogate Solution" into 100ml Hexane	Level 5 Conc. (ug/ml) [1] 34 ul Picloram Methyl ester stock (9000ug/ml) & 600 ul 'Surrogate Solution" into 100ml Hexane
Picloram	0.047	0.236	0.709	1.42	2.84
2,4-DCAA methyl ester (surr)	0.095	0.477	1.43	2.86	5.73

[1] The calculated final standard concentrations have been converted from the deriviatized concentration to the free acid equivalent concentration using the factors found in Attachment 1

7.3.3. Herbicide Spiking Standard (LCS/MS/MSD):

7.3.3.1 Herbicide QC Spiking Standard Mix (10 compound analysis): The ten (10) compound spiking standard is prepared and used when analyzing for an expanded list of herbicides. The 10 compound spiking mix is prepared using volumetric glassware as detailed in Table 7 (below) using a concentrated solution of free acid herbicides (Accustandard Catalog # M-1850A; see Tables 1 and 2):

Table 7: Preparation of Total Herbicide 10 Compound Spiking Mix	
Parameter	**Final Conc. (ug/ml)** 5.0 ml 'Spiking Mix -10 compounds)' into 10ml Acetone
2,4-D	50
2,4,5-T	50
2,4,5-TP (Silvex)	50
Dalapon	50
2,4-DB	50
Dicamba	50
Dichloroprop	50
Dinoseb	50
MCPA	5000
MCPP	5000

The concentrate is used for spiking of the following quality control sample types: MS, MSD, and LCS. For spiking instructions see the appropriate herbicide prep SOP referenced in Section 1.1.

7.3.3.2 **Pentachlorophenol QC Spiking Standard:** The pentachlorophenol spiking standard is prepared and used when analyzing for this herbicide. The pentachlorophenol spiking mix is prepared using volumetric glassware by diluting 1ml of the concentrated pentachlorophenol solution Spex # S-2950; see Tables 1 and 2) into a final volume of 10ml acetone. The concentrate is used for spiking of the following quality control sample types: MS, MSD, and LCS. For spiking instructions see the appropriate herbicide prep SOP referenced in Section 1.1.

7.3.3.3 **Picloram Acid QC Spiking Standard:** The picloram acid spiking standard is prepared and used when analyzing for this herbicide. The standard is prepared using a source containing the picloram free acid. Prior to the preparation of the picloram acid spiking standard a working stock solution must be prepared using the neat material described in Table 2. This working stock solution is prepared at 10000 ug/ml by dissolving 10.00 grams of neat picloram free acid into 1000 ml of hexane using class A volumetric glassware

7.3.3.3.1 Prepare the 5000 ug/ml picloram spiking solution by diluting 500ml of the working stock solution (see Sec. 7.3.3.4) into 1000 ml of hexane using class A volumetric glassware. For spiking instructions see the appropriate herbicide prep SOP referenced in Section 1.1

7.3.4. **Herbicide Surrogate Spiking Standard (**2,4-DCAA Acid Spike Mix**):** The herbicide surrogate spiking solution is a concentrated 10 ml solution of 2,4-DCAA at a concentration of 100 ug/ml. (Supelco Catalog # 861271, see Tables 1 and 2). The concentrate is used without further dilution prior to the spiking of samples.

7.3.5. **Initial Calibration Verification Standard:** The ICV is prepared as detailed in Section 7.3.2 (dependent upon the compound being analyzed) and must be from a separate lot or separate source from the standards used in the Initial Calibration Range (the analyst must confirm and document that a separate source or lot is used).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass	1L amber	Cool 4 ± 2ºC	7 days to extraction; 40 days to analysis	USEPA SW846
Soil/Sediment	Glass	4 oz wide mouth	Cool 4 ± 2ºC	14 Days to extraction; 40 days to analysis	USEPA SW846

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2ºC) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria. If the spiked sample recovery results fall outside the laboratory generated limits (refer to the current active TALS method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in 9.1.2.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing 2,4-DCAA (see Section 7.3.4). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.2 Instrument QC

9.2.1. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.1.1. ***Initial Calibration Range*:** A five-point calibration range is analyzed for each target compound using herbicide methyl ester standard mixes. Standards are prepared following the instructions in Section 7.3.

9.2.1.2. ***Initial Calibration Verification (ICV):*** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed.

The ICV is prepared as detailed in Section 7.3.2 (dependent upon the compound being analyzed) and must be from a separate lot or separate source from the standards used in the Initial Calibration Range (the analyst must confirm and document that a separate source or lot is used).

9.2.2. Continuing Calibration Verification (CCV): A mid-point Continuing Calibration Verification (CCV) must be analyzed every 12-hours or 20 samples (whichever is more frequent) and at the end of each analytical sequence. The CCV consists of the Level 3 calibration standard (~700 ppb) detailed in Section 7.3

9.2.3. Calibration Acceptance Summary

9.2.3.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. All gas chromatographs used for pesticides analysis at Eurofins Edison are equipped with electronic pressure control (EPC). The use of EPC results in little retention time variability between analyses. Accordingly, retention time variability for the purpose of retention time window determination for standards analysis is extremely small. The default retention time window option must therefore be employed as follows to accommodate the excellent precision of EPC equipped systems.

9.2.3.1.1 Obtain the retention time for all single component compounds from the analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. Apply the retention time window data in the table in Attachment 2 to its corresponding compound (ex., mid-point RT ±0.10 minute). Calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument.

9.2.3.1.2 New retention time windows must be established when whenever a chromatographic column is replaced or a new detector is installed .Whenever the observed retention time of each analyte and surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.

9.2.3.2. Initial Calibration Range: External standard calibration is employed for this method. The response factor (defined as the

ratio of the area to the standard concentration) is calculated for each analyte at each calibration concentration. The average response factor may be used for quantitation if the % RSD across the 5 point range is <20%. Alternatively, linear regression may be used if the correlation coefficient (r1) is ≥ 0.990 (note: if the linear regression model is used the curve must NOT be forced through the origin). Calibration is checked every 12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single response pesticide standards.

9.2.3.2.1 Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

RF = Response Factor

9.2.3.2.2 If the % RSD across the 5 point range is <20% the calibration can be assumed to be linear and the average response factor can be used to calculate concentrations of target compounds in samples.

9.2.3.2.3 If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be ≥ 0.990 for the calibration to be acceptable. Calibration is checked every 12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single component pesticide standards.

9.2.3.3. Relative Standard Error: The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation. The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r_2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point

9.2.3.4. Initial Calibration Verification (ICV): An ICV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed immediately after an initial calibration range. The calculated concentration of the ICV must be within ±15%D of the expected concentration. Should the %D exceed 15% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 15% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.

9.2.3.5. Continuing Calibration Verification (CCV): A CCV will consist of an ICAL standard at or near the midpoint of the Initial Calibration Range analyzed after each 10 samples (every 12 hours at minimum). The calculated concentration of the CCV must be within ±15%D of the expected concentration. Should the %D exceed 15% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 15% after a single CCV reinjection, a new Initial Calibration Range must be analyzed.

Step	*Standards*	*Type*	*Control Limit*	*Frequency*
Method # 8151A Instrument QC Summary				
Initial Calibration	*5 point*	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r ≥0.990 or <20%RSE*	*As required when ICV or CCV do not meet requirements*
ICV	*Separate source (midpoint)*	*Average*	*± 15%D*	*After every initial calibration*
CCV	*ICAL source (midpoint)*	*Average*	*± 15%D*	*1 in 10 or fewer samples (every 12 hours minimum)*

10.0 Procedure

10.1. Gas Chromatograph Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified.

Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. Injection System: A split/splitless injection port with electronic pressure control (EPC) is used. Thirty seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port. The EPC is used in constant flow mode.

10.1.2.1. For herbicide analysis the normal operating conditions of the injection port are as follows:

Injection port Temperature:	275°C
Temperature Rate:	10 deg C/minute
Inlet pressure	4.0 psi
Initial Temperature:	55 deg C
EPC:	Constant flow
Detector temperature	300C

10.1.2.1.1 For pentachlorophenol analysis the following injection port settings are used:

Injection port Temperature:	250°C
Temperature Rate:	4 deg C/minute
Inlet pressure	7.9 psi
Initial Temperature:	190 deg C
EPC:	Constant flow
Detector temperature	300C

10.1.2.2. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. The plug of glass wool is located in the liner between the double goose neck.

10.1.2.3. This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.4. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be

changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.5. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.6. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.7. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.3. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.3.1. The standard oven program for total herbicide analysis is :

Initial Temp	Time	Rate	Final Temp	Time
55°C	3.0 min	10°/min	244°C	11.5 min

10.1.3.2. The standard oven program for TCLP herbicide analysis is :

Initial Temp	Time	Rate	Final Temp	Time
190°C	3.0 min	4.0°/min	238°C	0.00 min

10.1.4. Detectors: Detectors operate at 300°C and need to be supplied with 40-60 ml/min total flow. They are essentially maintenance free on a day-to-day basis. They are routinely baked out at 325°C to remove persistent contaminants. On occasion the detectors may be baked out at a higher temperature to remove contaminants with an extremely high boiling point (CAUTION: Do not exceed the maximum detector temperature of 380°C).

10.1.4.1. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.5. Chemstation: The HP Chemstation application is used for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.6. Eurofins's Chrom chromatography data processing application is utilized for the processing of ChemStation chromatography data files. Calibrations, verification standards and samples are processed using this database. All of the processed files are imported into the TALS database. Data is processed and first level reviewed by the analysts. Senior analysts or managers do the second level review and update the status of the job to 'Lab Complete' in TALS. All client reports are generated in TALS.

10.1.7. Eurofins LIMS (TALS): All data files processed in Chrom are then imported to into TALS. The data files are reprocessed in TALS after linking the appropriate reagents and standards to the batch. All samples and quality control runs are reviewed. A TALS non-conformance memo (NCM) is written for any QC outlier or situation requiring additional documentation. Samples and QC results are assigned to primary/secondary column as per reporting requirements. Files that are not reported are marked rejected. All data files that are being reported are first level reviewed in TALS by the analyst. Second level TALS review is done by a trained peer or supervisor. Once second level review is completed in TALS reports are generated and reviewed after which the job is marked complete.

10.2. Analytical Sequences

10.2.1. Calibration Ranges:

10.2.1.1 Herbicides are calibrated using a five-point calibration range. Standards are prepared as detailed in Section 7.3 and analyzed under the instrumental conditions detailed in Section 10.1 as appropriate for total or TCLP herbicide analysis.

10.2.1.2 A calibration factor, defined as the ratio of the response to the amount injected, is calculated for each analyte at each standard concentration. The average calibration factor (if the %RSD across the five-point range is <20%) or a linear calibration not through the origin (if the correlation coefficient (r1) is $\geq$ 0.99) is used for quantitation. Calibration is

checked every 12 hours or after every ten (10) samples, whichever comes first, by injection a calibration check standard.

10.2.1.3 Generating a calibration table: After the five calibration standards have been run, the data files are copied over to the Chrom data system. A new Chrom batch is created and the method file from the previous Chrom batch is copied to the new Chrom batch.

10.2.1.4 The data files representing the five levels of calibration are also copied to the new Chrom batch and processed with the method file. The integration of the five levels is checked for consistency in Chrom Review.

10.2.1.5 Linear Calibration. Check the percent relative standard deviation (% RSD) of the calibration factors for each individual analyte. If the % RSD is less than 20% over its working range, the linearity of the range is assumed. The average value of the calibration factors is used for quantitation of all the samples and continuing standards.

10.2.1.6 Linear Calibration Using Least Squares Regression. If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be $\geq$ 0.99 for the calibration to be acceptable.

10.2.1.7 Since the calibration is performed using standards made from methyl ester compounds (compounds not esterified by application of this method), then the calculation of free acid herbicide concentrations must include a correction for the molecular weight of the methyl ester versus the acid herbicide (see Attachment 1).

10.2.2. Calibration Check Standards:

10.2.2.1. **Initial Calibration Verification** (ICV) is analyzed and evaluated immediately after the initial calibration range as detailed in Sections 7.3 (standard prep) and 9.2 (instrument QC).

10.2.2.2. **Continuing Calibration Verification** (CCV): is analyzed and analyzed after each 10 sample injections as further detailed in Sections 7.3 (standard prep) and 9.2 (instrument QC). The CCV is also analyzed at the end of each analytical sequence as a closing calibration check standard.

10.2.2.3. Note that samples are always quantified against the average calibration factor from the initial calibration range (or from the initial calibration curve). Calibration check standards serve only

to confirm the validity of the initial calibration range and are never used to quantify sample concentrations.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	≤20% RSD
Cont. Cal. Verif. (CCV)	Prior to / after every 10 injections	Prior to/after every 10 injections
RT Windows (RTW)	Init. CCV determines midpt. of RTW	±3X SD

10.2.3. Idealized Analytical Sequence

10.2.3.1. Analysis Sequence: The automation of the GC runs is accomplished via the "SEQUENCE" macro of the Chemstation.

10.2.3.2. The Sequence File: The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle

10.2.3.3. It is common practice to evaluate the instrument status, and then complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro.

10.2.3.4. An idealized analytical sequence is presented below:

Ideal Analysis Sequence*
Instrument Blank (solvent injection)
Initial Calibration Range Standards (5)
ICV
CCV
Method Blanks and LCS
Client Samples (10)
CCV
Client Samples (10)
CCV*

*After the analysis of ten sample injections or 12 hours of analysis, a CCV must be successfully analyzed.

10.3. Documentation

10.3.1. After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor, method, job

number, QC Batch number, extraction date, lab prep batch, Chrom batch, signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (e.g., 'O.K'., 're-run at dilution', 'non-inject', etc.).

10.4. Dual Column Approach

10.4.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. If the difference between the dual columns results in ≤40% RPD report the higher concentration.

10.4.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows established from the most recent preceding calibration verification.

10.4.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P.

10.4.4. If the surrogates on one column are very different (>40% RPD) compared to the other column, this may be indicative of a bad injection or column blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.4.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.4.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.4.6.1 An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

10.4.7 In summary, the flow chart in Attachment 3 presents a recommended rational approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.5. Data Processing

10.5.1. Process all samples, QC samples, and blanks using Chrom. Evaluate the QC samples (Method Blank, MS/MSD, and LCS) against the criteria detailed in Section 9.1. Take corrective action as detailed in that section.

10.5.2. Evaluate chromatograms for carryover and cross-contamination. Any samples suspected of having carryover must be re-prepped and re-analyzed. To make sure there is no carryover, reagent water is injected before the re-prepped sample.

10.5.3. Any compound concentration that exceeds the concentration of the calibration range must be diluted and re-analyzed.

10.5.4. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and %moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. $$\text{Concentration = mg/kg or L} = \frac{C \times V \times D}{W}$$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared

11.4. Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i-1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level i

.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification:

The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.4. Training Requirements

Refer to Eurofins Edison SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

The following waste streams are generated when using this method:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the

waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

Teris Profile Number: 50016624
Onyx Profile WIP Number: 545240

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method 8151A: Organochlorine Herbicides by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. Eurofins Edison SOP No. ED-ORP-015, Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A, current revision.

15.3. Eurofins Edison SOP No. ED- ORP-023,Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A, current revision.

15.4. Eurofins Edison Document No. ED-QA-LQM, Laboratory Quality Manual, current revision.

15.5. Eurofins Corporate Document No. CW-E-M-001, Environmental Health and Safety Manual, current revision.

15.6. Eurofins Corporate Quality SOP No. CA-Q-S-001, Solvent & Acid Lot Testing & Approval, current revision.

15.7. Eurofins Edison SOP No. ED-GEN-023, Bulk Solvent Testing and Approval, current revision.

15.8. Eurofins Edison SOP No. ED-GEN-013, Glassware Cleaning, most current revision

15.9. Eurofins Edison SOP No. ED-GEN-022, Training, current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

Attachment 1: Herbicide Free Acid Conversion Chart

Attachment 2: Retention Time (RT) Windows for Primary Herbicides/Surrogate

Attachment 3: Dual Column Reporting Flowchart

18.0. Revision History

- Revision 12, dated 17 Oct 2022
    - Updated throughout to reflect Eurofins branding.
    - Section 9.2.3.3: Included details of evaluation of initial calibration for Relative Standard Error (RSE).
    - Section 11.4: added formula for calculation of RSE.
    - Section 12.2 added: details the annual LLOQ verification requirement.

- Revision 11, dated 22 March 2021
    - Added Section 10.5.4 which details the establishment of the reporting limit (RL).

- Revision 10, dated 10 December 2020
    - Updated throughout to reflect Eurofins branding.
    - Revised references to Quality Manual section numbers as required

- Revision 9, dated 22 February 2016
    - Revised throughout to correct the spelling of Picloram (4-amino-3,5,6-trichloropicolinic acid).

- Revision 8, dated 12 August 2013
    - Revised throughout to reflect the lab's implementation of the TestAmerica Chrom data system.
    - Deleted Section 7.3.2.1 (Total Herbicide Calibration Standards, 3 Compound Analysis). Renumbered remaining sections accordingly.
    - Deleted Section 7.3.1.1 (Herbicide QC Spiking Standard, 3 Compound Analysis). Renumbered remaining sections accordingly.
    - Deleted Table 4 (Preparation of Total Herbicide Calibration Standards, 3 Compound Analysis). Renumbered remaining tables accordingly.
    - Revised 2,4,5-TP (Silvex) methyl ester concentrations (all levels) and Dalapon (level 3) in Table 4 (formerly Table 5)
    - Revised Pentachlorophenol concentrations (levels 2 and 3) in Table 5 (formerly Table 6)
    - Revised Picloram concentrations (all levels) and 2,4-DCAA methyl ester (level 4) in Table 6 (formerly Table 7)

- Revision 7, dated 15 August 2011
    - Section 7.3.3.4: Corrected weight of neat Picloram from 9 grams to 10 grams
    - Attachment 3: revised to reflect current practice.
    - Revised 'Distribution' footer on Cover Page to reflect current practice.

- Revision 6, dated 28 October 2009

    - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
    - Section 1.1. Added Table 1, *Analytes and Reporting Limits*
    - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
    - Section 1.1: Expanded to include references to applicable prep and cleanup SOPs.
    - Section 3: revised to reference new location for definitions.
    - Section 5: Revised to include most up to date corporate health and safety references and information.
    - Section 7.2.2: Added table detailing components found in the various standards mixes.
    - Section 7.3: Updated the instructions for preparation of standards.
    - Section 7.3: Added tables with calibration standards prep details.
    - Section 8: Updated with additional details including a table outlining containers, preservation and holding times for waters and soils.
    - Section 9.1: Expanded QC sample preparation, analysis, evaluation and corrective action details.
    - Section 9.2: Expanded details of preparation, analysis, evaluation and corrective action for initial and continuing calibration and calibration verifications.
    - Section 10.2.3: added procedures for dual column evaluation.
    - Section 10.4.1: Updated Idealized Analytical Sequence table.
    - References: Expanded to include more specific SOP references
    - Section 18: Added this Revision History section
    - Throughout document: added references to TestAmerica LIMS (TALS).
    - Attachments: Added Attachment 3: Dual Column Reporting Flowchart

Attachment 1

Herbicide Free Acid Conversion Chart

Analyte	Free Acid Molecular Weight	Methyl Ester Molecular Weight
Dalapon	142.97	156.97
2, 4 - D	221.00	235.00
2, 4, 5 - TP (Silvex)	269.50	283.50
2, 4, 5 - T	255.50	269.50
Dinoseb	240.22	254.22
2, 4 - DB	249.09	263.09
Dicamba	221.04	235.04
Dichloroprop	235.05	249.05
Picloram	241.48	255.48
Pentachlorophenol	266.50	280.50
MCPA	200.63	214.63
MCPP	200.63	214.63
DCAA	297.13	311.13

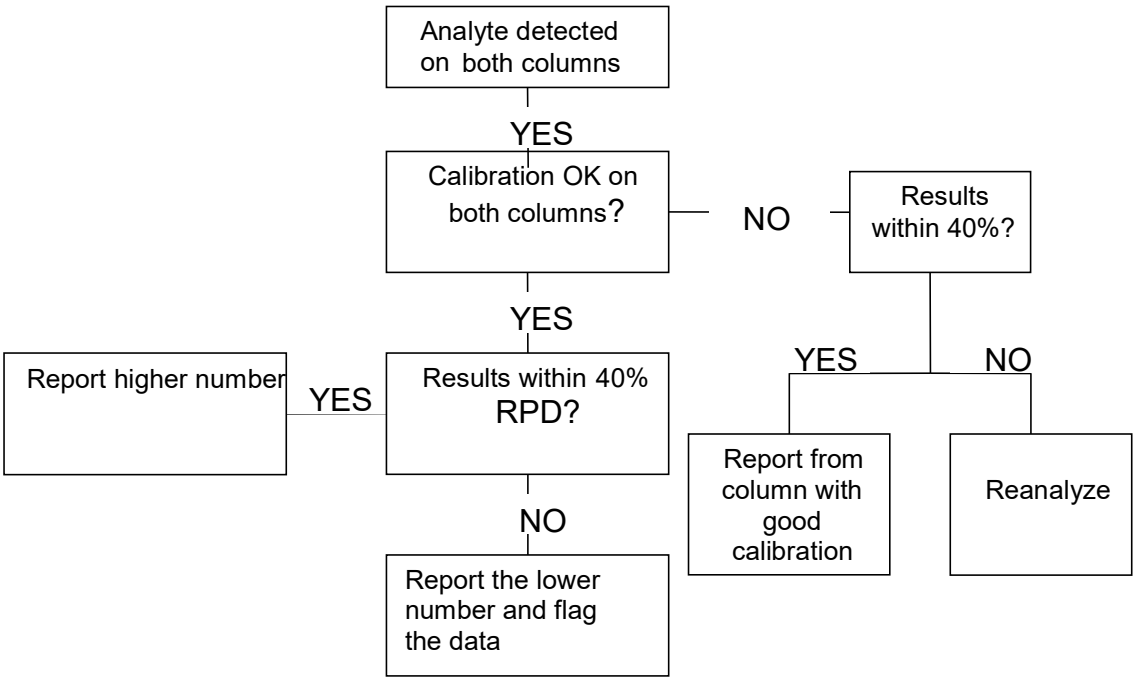
$$[\text{Free Acid}] = \frac{[\text{Methyl Ester}] \times \text{Free Acid Molecular Weight}}{\text{Methyl Ester Molecular Weight}}$$

Attachment 2

Retention Time (RT) Windows For Herbicides/Surrogate

Compound	RT Window (minutes)	
	Total Herbs	TCLP Herbs
2,4-D	*± 0.10*	*± 0.05*
2,4,5-TP (Silvex)	*± 0.10*	*± 0.05*
2,4,5-T	*± 0.10*	*± 0.05*
Dalapon	*± 0.10*	
2,4-DB	*± 0.10*	
Dicamba	*± 0.10*	
2,4-DP (Dichloroprop)	*± 0.10*	
Picloram	*± 0.10*	
Pentachlorophenol	*± 0.10*	
MCPA	*± 0.10*	
MCPP	*± 0.10*	
Picloram	*± 0.10*	
DCAA (surrogate)	*± 0.10*	*± 0.10*

Attachment 3
Dual Column Reporting Flowchart



Title: Trace Metals Analysis for Water, Wastewater, Soil and Sediment Samples by ICP-MS EPA Method 200.8

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Approvals (Signature/Date):

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	01/15/2023		01/13/2023
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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes the procedures used to determine the concentration of various elements in surface, drinking, and ground water, wastewater, by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) using EPA Method 200.8.

The routine target analytes and reporting limits are as follows:

Table 1

Analyte List and Method Reporting Limits (RL)

Element	Drinking Water RL	Non-Drinking Water RL
	(ug/L)	200.8 NPDES (ug/L)
Aluminum (Al)	50	40
Antimony (Sb)	0.4	2
Arsenic (As)	1	2
Boron (B)	20	80
Barium (Ba)	2	4
Beryllium (Be)	0.3	0.8
Cadmium (Cd)	0.5	2
Calcium (Ca)	250	500
Cobalt (Co)	2.5	4
Chromium (Cr)	0.9	4
Copper (Cu)	2.5	4
Iron (Fe)	250	120
Lead (Pb)	2.0	1.2
Magnesium (Mg)	250	200
Manganese (Mn)	25	8
Molybdenum (Mo)	2.5	4
Nickel (Ni)	0.5	4
Potassium (K)	250	200
Selenium (Se)	8	2.5
Silver (Ag)	2.5	2
Sodium (Na)	250	500
Strontium (Sr)	2.5	4
Thallium (Tl)	0.3	0.8
Tin (Sn)	4	16
Titanium (Ti)	2.5	4
Vanadium (V)	2.5	4
Zinc (Zn)	20	16

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Prior to analysis by ICP-MS, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

For total recoverable analysis of aqueous samples containing undissolved material and for drinking water samples with turbidity $\geq$ 1 NTU, samples must undergo digestion prior to analysis. Refer to TestAmerica Edison SOP Nos. ED-MTP-001. For the determination of dissolved analytes in a filtered aqueous sample and in a drinking water sample with turbidity < 1 NTU, samples don't require digestion prior to analysis. Samples are ready for analysis after the addition of nitric acid.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. Isobaric polyatomic/molecular ion interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest and which cannot be resolved by the mass spectrometer in use. Most isobaric interferences that could affect the ICP-MS analysis for elements in this SOP have been identified. These can be managed by the selection of an alternate isotope, the use of the collision/reaction cell (used for non-potable samples only), or by the use of elemental interference equations when the collision/reaction cell is not in use.

4.2. For Drinking Water analysis (standards, samples, and relating QC) the collision/reaction cell can not be used; therefore, the following interference equations should be used to reduce interferences:

Mass	Equation
6:	(6)*1 - (7)*0.082
44:	(44)*1 - (88)*0.015
51:	(51)*1 – (53)*3 + (52)*0.34

66: (66)*1 – (69)*0.00141
75: (75)*1 – (77)*2.9 + (82)*2.23 – (83)*2.23
82: (82)*1 – (83)*1
111: (111)*1 – (108)*1.073 + (106)*0.712
115 (115)*1 – (118)*0.016
208: (208)*1 + (206)*1 + (207)*1 (used for both drinking and non-drinking water analysis)

The basic elemental interference equations are based on natural isotopic abundances. The most precise coefficients for an instrument must be determined from the ratio of the net isotope signals that are observed for a known standard solution at a concentration sufficient to produce suitable counting statistics.

4.3. Krypton affects the determination of both arsenic and selenium but can be greatly reduced with the use of high purity Krypton free argon. Krypton must be analyzed for every sample and standard.

4.4. Physical interferences are effects associated with the sample nebulization and transport processes as well as ion-transmission efficiencies. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. These changes in matrix can cause significant signal suppression or enhancement. Dissolved solids can deposit on nebulizer tips and interface cones (reducing the orifice size and the instrument's performance). Internal standards can be used to correct for physical interferences if they are carefully matched to the analyte so that both elements react similarly to the matrix changes.

4.5. Memory interferences can occur when analytes from a previous sample contribute to signals measured from subsequent samples. The memory effects can result from analyte deposition of sample on the sample tubing, joints, nebulizer, spray chamber, torch, and/or interface cones. Routine maintenance on the sample introduction system is necessary in order to minimize the memory interferences. The memory effects must be taken into account when setting up a suitable rinse times. The evaluation of a minimum of three replicate integrations will help to determine memory problems.

4.6. Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Agilent 7800 and Agilent 7900: inductively coupled plasma mass spectrometer with data system (microprocessor, monitor, printer) and autosampler. Mass Range 2-260 amu. Vacuum purged spectrophotometer with an axial plasma torch. Collision/Reaction Cell.

- Heat exchanger –PolyScience Model 6106T Recirculating Chiller or equivalent
- Autosampler – Agilent SPS 4 Autosampler

6.2. Supplies

- Reagent Water -18 megohm Reagent grade Type II water
- Volumetric Flasks (Class A): 50 mLs, 100 mLs, 500mls & 1000mls
- Eppendorf & Fisher Pipettes, varying volumes
- Polypropylene tubes

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Concentrated Nitric Acid (HNO3) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.2. Concentrated Hydrochloric Acid (HCL) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.3. Argon supply - 99.9% (Liquid)

7.1.4. Helium and Hydrogen supply – 99.999% (Gas)

7.1.5. 18 megohm Reagent grade Type II water.

7.1.6. Reagent water, 2% HNO3 + 0.5% HCl by volume: Add 200ml of concentrated HNO3 and 50ml of concentrated HCl to deionized water and bring to 10 liter volume with deionized water. For potable samples not requiring digestion, the reagent water is prepared without HCL. Note: Always add acid to water. Record preparation in the TALS Reagent Module Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature.

7.1.7. 5% HNO3 + 5% HCl: Add 1 liter of concentrated HNO3 and 1 liter of concentrated HCl to deionized water and bring to 20 liter volume with deionized water. For potable samples not requiring digestion, the reagent water is prepared without HCl. Varying amounts of this reagent may be made (e.g., 10L) by proportionally adjusting the volume of acids used.

Note: Always add acid to water. Record preparation in the TALS Reagent Module Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature.

7.2. Standards

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Certificates of analysis or purity must be received with all neat compounds or stock solutions. Certificate of analysis are filed in Metals managers office.

Storage requirements: all standards are stored at room temperature

Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – 6 months
Working cal standards – two weeks
Working Initial calibration verification standard – quarterly
Interference Check Standards (A&B) - weekly
(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: Final concentrations for the calibration standards are given in Attachment 1. All standards must be prepared in reagent water (Sec. 7.1.6 & 7.1.7).

Standards must be prepared in accordance to the shelf life (Sec 7.2) "or sooner if needed or required." "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

All standards must be prepared in reagent water (Sec. 7.1.6 and 7.1.7). Final concentrations for the calibration standards are given in Attachment 1. Varying amounts of these standards may be made by proportionally adjusting the volume of standards and reagent water used.

7.2.1. Calibration Standards: ICPMS-Cal (Custom Calibration Standard Stock Solution) purchased from Inorganic Ventures and from CPI International. Refer to manufacturer's instructions for stability and storage information.

6020B RL Standard contains: 20 mg/L Ca, K, Mg, Na
12 mg/L Fe
8 mg/L B
4 mg/L Al
1.6 mg/L Sn, Zn
0.8 mg/L Mn
0.4 mg/L Ba, Co, Cr, Cu, Mo, Ni, Sr, Ti, V
0.25 mg/L Se
0.2 mg/L Ag, As, Cd, Sb,
0.12 mg/L Pb
0.08 mg/L Be, Tl

TA-CAL-1 contains: 1,000 mg/L Si
100 mg/L As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V

TA-CAL-2 contains: 1,000 mg/L Al, Ca, Fe, K, Mg, Na

Single Element Standard: Ag 1,000 mg/L

Single Element Standard: B 1,000 mg/L

Single Element Standard: Zn 1,000 mg/L

200.8 Potable RL Std contains: 25000 ug/L Ca, Fe, K, Mg, Na
2500 ug/L Al, Mn,
2000 ug/L B
800 ug/L Se
400 ug/L Sn, Zn,
250 ug/L Ag, Co, Cu, Mo, Sr, Ti, V
200 ug/L Ba, Pb
100 ug/L As
90 ug/L Cr
50 ug/L Cd, Ni
40 ug/L Sb
30 ug/L Be, Tl

7.2.2. Working Calibration Standards:

For Non-Drinking Waters:

CAL5: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 0.4ml of TA-CAL-1 and 5ml of TA-CAL-2. Add 10 ml of MS-Cal-Int. Bring to volume with 5% HNO3 / 5% HCl.

CAL4/CCV: Add 10ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 50ml polypropylene tube. Add 10ml of working Cal5 and mix.

CAL3: Add 15ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 50ml polypropylene tube. Add 5ml of working Cal5 and mix.

CAL2: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 10ml of 6020B RL Standard. Bring to volume with 5% HNO3 / 5% HCl.

CAL1: Add 10ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 50ml polypropylene tube. Add 10ml of working Cal2 and mix.

MS-Cal-Int: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 2ml each of B and Zn. Add 0.4ml of Ag. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7).

For Drinking Waters:

CAL5: Add 100ml of reagent water (Sec 7.1.6) to a clean 200ml flask. Add 0.2ml TA-CAL-1, 0.5ml of TA-CAL-2, 0.04ml of 1000ppm B, and 0.02ml each of 1000ppm Ag & Zn. Bring to volume with reagent water (Sec 7.1.6).

CAL4/CCV: Add 10ml of reagent water (Sec 7.1.6) to a clean 50ml polypropylene tube. Add 10ml of Cal5 (drinking water) and mix.

CAL3: Add 15ml of reagent water (Sec 7.1.6) to a clean 50ml polypropylene tube. Add 5ml of Cal5 (drinking water) and mix.

CAL2: Add 18ml of reagent water (Sec 7.1.6) to a clean 50ml polypropylene tube. Add 2ml of Cal5 (drinking water) and mix.

CAL1: Add 100ml of reagent water to a clean 200mL flask. Add 2 mL of the 200.8 Potable RL Std. Bring to volume with reagent water (Sec 7.1.6). This standard is used as the Reporting Limit standard (CRI).

Note: see Attachment 1 for final elemental concentrations.

7.2.3. Initial Calibration Verification Stock standards: purchased from CPI International. Refer to manufacturer's instructions for stability and storage information. Standards must be from a different source than those used for the calibration standards.

TA-CAL1-SS contains: 1,000 mg/L Si
100 mg/L As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V

TA-CAL2-SS contains: 1,000 mg/L Al, Ca, Fe, K, Mg, Na

2nd Source Single Element Standard: Ag 1,000 mg/L

2nd Source Single Element Standard: B 1,000 mg/L

2nd Source Single Element Standard: Zn 1,000 mg/L

7.2.4. ICV Working Standard:

7.2.4.1. ICV Potable: The ICV Working Standard must be at a concentration different from the concentrations used for the CCV working standard. Prepare by adding 100ml of reagent water (Sec 7.1.6) to a clean 500ml flask. Add 0.2ml TA-CAL-1-SS, 0.5ml of TA-CAL-2-SS, 0.04ml of 1000ppm B (2nd source std), and 0.02ml each of 1000ppm Ag & Zn (both 2nd source std). Bring to volume with reagent water (Sec 7.1.6). The final concentrations of the various elements are listed in Attachment 1. This standard is prepared fresh quarterly or as needed and stored at room temperature.

7.2.4.2. ICV Non-Potable: The ICV Working Standard must be at a concentration different from the concentrations used for the CCV working standard. Add 10ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 50ml polypropylene tube. Add 10ml of working Interm_ICV (Sec 7.2.4.2.2) and mix. The final concentrations of the various elements are listed in Attachment 1. This standard is prepared fresh quarterly or as needed and store at room temperature.

7.2.4.2.1. MS-ICV-Int: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 2ml each of 2nd Source: B & Zn. Add 0.4ml of 2nd Source: Ag. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7).

7.2.4.2.2. Interm ICV: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 0.32ml of TA-CAL-1-SS and 1.6ml of TA-CAL-2-SS. Add 8 ml of MS-ICV-Int (see Sec 7.2.4.2.1). Bring to volume with 5% HNO3 / 5% HCl

7.2.5. Continuing Calibration Verification standard, Non-potable: see CAL4/CCV (Sec. 7.2.2).

7.2.6. Continuing Calibration Verification standard, Potable: see CAL4/CCV (Sec. 7.2.2).

7.2.7. Internal Standards and EPA Tune Check:

The following elements are used as the internal standards: Bi, In, Li6, Sc, Tb, and Ge.

7.2.7.1. Internal Standards Stock Solutions**:**

7.2.7.1.1. Testamerica-1 (Custom Calibration Standard Stock Solution) purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information. Contains: 250 mg/L each: Li6, Sc; 50 mg/L each: Bi, In, Tb.

7.2.7.1.2. MSGE-100PPM (Custom Calibration Standard Stock Solution) purchased from CPI International. Refer to manufacturer's instructions for stability and storage information. Ge 100 mg/L.

7.2.7.1.3. Triton X-100 (Standard Stock Solution: Non-ionic Surfactant) purchased from Fisher Scientific, p/n BP151-100. Refer to manufacturer's instructions for stability and storage information. According to the ICPMS manufacturer (Agilent Technologies), Triton-X 100 will reduce the 3 replicate %RSD during analysis.

7.2.7.2. Working Internal Standard Solution: *For Non-Potable*: Add 100ml of reagent water to a clean 1000ml flask. Add 1 ml of Testamerica-1 and 1 ml of MSGE-100PPM, and 0.1 ml of Triton X-100. Bring to volume with reagent water. *For Potable*: Add 100ml of reagent water to a clean 250ml flask. Add 0.5 ml of Testamerica-1, 0.5 ml of MSGE-100PPM, and 0.05 ml of Triton X-100. Bring to volume with reagent water.

The following are the recommended analysis masses, tune steps, and internal standards:

Element	IS
Be9	Li6
B11	Li6
Na23	Sc45
Mg24	Sc45
Al27	Sc45
K39	Sc45
Ca40	Sc45
Ti47	Sc45
V51	Sc45
Cr52	Sc45
Mn55	Sc45
Fe56	Sc45
Co59	Sc45
Ni60	Sc45
Cu63	Sc45
Zn 66	Sc45
As75	Sc45
Se78	Sc45
Sr88	In115
Kr83**	(none)
Mo95	In115
Ag107	In115
Cd111	In115
Sn118	In115
Sb121	In115

Ba137	Tb159
Tl205	Bi209
Pb 208*	Bi209

*Pb 208 = Pb 208 + Pb 207 + Pb 206
**Kr83 is used for monitoring Krpyton levels only.

Different masses and internal standards may be utilized, as matrix issues deem necessary. Non-drinking waters are analyzed using collision/reaction cell technology (helium/hydrogen gas mode). Typical non-drinking water Collision/Reaction Cell usage: Calcium, Iron, and Selenium are analyzed in the Hydrogen reaction mode; Beryllium and Boron Titanium are analyzed in the No Gas mode; all other elements are analyzed in the helium collision mode. For drinking water analyses, collision cell technology is not allowed; therefore, the no-gas mode is utilized. Use masses Ca44, Fe57, Cu65, and Se82 instead of their respective masses listed above.

Tune check criteria:

Instrument tunes must be performed daily, before calibration. A solution at ~10ppb of Be, Mg, Co, In, and Pb is analyzed, and the precision, mass calibration, and resolution are checked. Resolution is checked by analyzing Magnesium isotopes 24, 25, 26 and Lead isotopes 206, 207, 208.

The following limits are used to evaluate mass calibration, resolution, and instrument stability:

The following limits are used to evaluate the tune:

Mass calibration: +/-0.1 amu
Resolution check: ≤0.9 amu at 10% peak height. Note: This also satisfies Method 200.8, 1.0 amu at 5% peak height)
Stability (5reps): <5%

7.2.7.3. EPA Tune Check Stock Standard: 2008TS purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information. Contains: 10 ug/mL each: Be, Co, In, Mg, Pb.

7.2.7.4. Working EPA Tune Check Standard: add 100ml of reagent water to a clean 1000ml flask. Add 1 mL of 2008TS. Bring to volume with reagent water. This solution contains 10 ug/L of Be, Co, In, Mg, and Pb

7.2.8. Reporting Limit (CRI) Check standard:

For Non-Drinking Water: See Section 7.2.2, CAL1
For Drinking Water: See Section 7.2.2, CAL1.

7.2.9. ICP Interference Check Solutions: Stock solutions for ICSA and ICSAB, purchased from Inorganic Ventures (6020ICS-8A & 6020ICS-8B) and CPI International (TA-ICPMS-ICSA & TA-ICPMS-ICSAB), contains the following elemental concentrations:

TA-ICPMS-ICSA contains: 10000 mg/L Chloride; 2000 mg/L Carbon; 1000 mg/L ea: Al, Ca, Fe, K, Mg, Na, P, S; 20 mg/L ea: Mo, Ti

TA-ICPMS-ICSAB contains: 100 mg/L W; 10 mg/L ea: As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Se, Si, Sn, Sr, V; 5 mg/L ea: Sb, Tl;

Single Element Standard: Ag 1,000 mg/L

Single Element Standard: Zn 1,000 mg/L

7.2.10. Working Interference Standards (ICSA/ICSAB):

For Non-Drinking Waters:

ICSA: Add 50ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 100ml flask. Add 10ml of TA-ICPMS-ICSA stock solution. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

ICSAB: Add 50ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 100ml flask. Add 10ml of TA-ICPMS-ICSA, 2 ml of TA-ICPMS-ICSAB, and 0.02 ml each of 1000 mg/L Ag and Zn. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.7). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

For Drinking Waters:

ICSA: Add 50ml of reagent water (Sec 7.1.6) to a clean 100ml flask. Add 2.5ml of TA-ICPMS-ICSA stock solution. Bring to volume with reagent water (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

ICSAB: Add 50ml of reagent water (Sec 7.1.6) to a clean 100ml flask. Add 2.5ml of TA-ICPMS-ICSA, 0.5ml of TA-ICPMS-ICSAB, and 0.005ml each of 1000 mg/L Ag and Zn. Bring to volume with reagent water (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

7.2.11. ICP-MS Matrix Spiking Solution, ICPMS LCS/SPK: purchased from High-Purity Standards. Refer to manufacturer's instructions for stability and storage information. Refer to attachment 2 for final elemental concentrations. Stock solution contains the following elemental concentrations:

Element	Conc. Of Stock (mg/L)
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	100
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	50

8.0. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size[2]	Preservation	Holding Time[1]	Reference

Matrix	Sample Container	Min. Sample Size[2]	Preservation	Holding Time[1]	Reference
Metals Waters except Hg	Polyethylene or Glass	250 mLs	HNO_3 to pH < 2 prior to shipment; if not, acidify upon receipt in lab [3,4]	6 months	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.
[2] Drinking water samples may require 1000ml sample volume.
[3] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥2, repeat steps (i.e., add acid, hold for 24 hrs, verify ph<2).
[4] Aqueous samples may be stored at room temperature.

Note: If drinking water samples are properly preserved with nitric acid to a pH<2 and the turbidity is <1 NTU, then acid digestion is not required. The laboratory must maintain documentation of the turbidity reading.

Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered. Properly filtered and nitric preserved samples requiring analysis for dissolved analytes don't require digestion prior to analysis.

9.0. Quality Control

Note: If a batch of samples requires digestion, then the relating QC samples must be carried through the entire digestion process.

9.1. Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Control	Frequency	Control Limit
Preparation blank	One per batch of twenty samples or less	**200.8**: < RL or <10% sample concentration.
Lab Control Sample Water (LCSW)	One per batch of twenty samples or less	**200.8**: +/- 15%
Matrix Duplicate	One in 20 or fewer samples	20% RPD
Matrix Spike (200.8)[1]	One, if 10 or less samples in batch; two, if 11 or more samples	70-130%
Matrix Spike Duplicate	When requested by client	70-130%; If MS and MSD are both ≥ 5X CRDL, then 20% RPD. If MS and MSD

Quality Control	Frequency	Control Limit
		are less than the CRDL, the RPD is not calculated; otherwise ±CRDL.
Post Digestion Spike	One per batch of twenty samples or less	See Section 9.1.6.4

[1] The sample for MS is randomly selected, unless specifically requested by a client.

9.1.1. Preparation Blank/Method Blank: One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. results must be less than the reporting limit (RL) or less than 10% of the determined analyte concentration for a sample. If any analyte concentration in the blank is above the control limit, the batch must be prepared again for the element in question and the samples reanalyzed.

9.1.2. Laboratory Control Sample Water (LCS): A laboratory control sample must be analyzed with each group of samples digested. Refer to Water Matrix Spike concentration in Attachment 1 for final elemental concentrations. Results must be within the acceptable control limits, if not, all samples prepared in association with the LCS must be redigested and reanalyzed.

- Method 200.8: wastewater samples - prepare the LCS by adding 0.25 ml of ICP-MS LCS/SPK (Sec. 7.2.11) in 50 ml deionized water. Results must be within ± 15% of the true value.
- Method 200.8: drinking water samples - prepare the LCS by adding 0.10 ml of ICP-MS LCS/SPK (Sec. 7.2.11) in 50 ml deionized water. Results must be within ± 15% of the true value.
- For dissolved aqueous samples not undergoing a preparation procedure, the CCV may serve as the LCS.

9.1.3. Matrix Duplicate: A duplicate is analyzed for each batch of samples digested. The relative percent difference between the results of the sample and the duplicate must fall within 20% RPD for samples greater than five times the reporting limit.

9.1.5. Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples digested.

- For aqueous MS sample, spike 0.25 ml of ICP-MS LCS/SPK (Sec 7.2.11) into 50 ml sample.
- For drinking water MS sample spike 0.10 ml of ICP-MS LCS/SPK into 50 ml sample.
- Method 200.8: a recovery of 70-130% is required.
  An exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more. If the recovery is not within specified limits a post digestion spike is required to be analyzed

at a concentration between 10 to 100 times the instrument detection limit. If the Post digestion spike recovery is not recovered within 85-115% for 200.8, a matrix effect should be suspected. See Attachment 1 for final Matrix spike concentration for water and soil.

9.1.6. Post-Digestion Spike: To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch). Post-Digestion Spike sample should be evaluated if serial dilution fails.

9.1.6.1. Transfer 10mL of a digestate to a suitable vial.

9.1.6.2. For Non-Potable samples, spike the sample with 0.05ml of ICP-MS LCS/SPK. For Potable samples, spike the sample with 0.01ml of ICP-MS LCS/SPK. Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed). The final concentration of the post digestion spike is as follows:

Element	Non-Potable Post Spike concentration (ug/L)	Potable Post Spike concentration (ug/L)
Aluminum (Al)	2500	500
Antimony (Sb)	25	5
Arsenic (As)	50	10
Barium (Ba)	50	10
Beryllium (Be)	25	5
Boron (B)	100	20
Cadmium (Cd)	25	5
Calcium (Ca)	2500	500
Chromium (Cr)	50	10
Cobalt (Co)	25	5
Copper (Cu)	50	10
Iron (Fe)	2500	500
Lead (Pb)	25	5
Magnesium(Mg)	2500	500
Manganese (Mn)	250	50
Mercury (Hg)	2.5	0.5
Molybdenum (Mo)	50	10
Nickel (Ni)	50	10
Potassium (K)	2500	500
Selenium (Se)	50	10
Silver (Ag)	25	5
Sodium (Na)	2500	500
Strontium (Sr)	50	10
Thallium (Tl)	20	4
Tin (Sn)	50	10

Element	Non-Potable Post Spike concentration (ug/L)	Potable Post Spike concentration (ug/L)
Titanium (Ti)	50	10
Vanadium (V)	50	10
Zinc (Zn)	50	10

9.1.6.3. Calculate the percent recovery of the post digestion spike as follows:

$$\%REC = \frac{Cps - Cs}{C2} \otimes 100$$

Where: Cps = concentration of post digestion spike (ug/L)
Cs = concentration of un-spiked sample (ug/L)
C2 = concentration of spike (ug/L

9.1.6.4. Limits for post digestion spike recoveries: 85-115%. Results must be within the acceptable limits, if not, repeat analysis and/or re-prepare the sample and reanalyze. If necessary dilute the QC sample (DU, MS, and PDS) to compensate for the matrix effect and reanalyze.

The post digestion spike must not be applied to samples analyzed at a dilution that produces a significant negative response. The analyst must use good judgment when evaluating data where the sample response is negative. Where a significant negative response is present, the digestate should be diluted and reanalyze to determine the extent of the matrix interferences. If necessary, adjust the interference corrections and reanalyze the samples.

9.1.7. Matrix Spike Duplicate (MSD): A matrix spike duplicate is prepared and analyzed when requested by the client. See Sec 9.1.5 for spiking procedures and acceptance limits.

- If matrix spike sample and matrix spike duplicate are both ≥ CRDL, then 20% RPD. If matrix spike sample and matrix spike duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.2. Instrument QC

Table 2 below describes the frequency, criteria, and corrective actions for the calibration and Quality control samples.

Table 2: Calibration, Quality control and Corrective Action Summary

QC Item	Frequency	Criteria	Corrective Action
Tune validation-Mass Calibration	Daily	Within 0.1amu from mass unit	Terminate analysis, fix problem and repeat
Tune validation-Resolution Check	Daily	≤0.9amu full width at 10% peak height	Terminate analysis, fix problem and repeat
Instrument Stability Check	Daily	<5% RSD for five replicates	Terminate analysis, fix problem and repeat
Initial Calibration: Multi-point-minimum 5 stds and 1 blank	Daily	Correlation ≥0.995	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	+/- 10%	Recalibrate
Continuing Calibration Verification Standard (CCV)	At the beginning and end of the analysis, and every 10 samples	Within +/- 10% of the true value	Fix the problem and reanalyze the previous 10 samples.
Calibration Blank (ICB/CCB)	After ICV and every CCV	Absolute value of the calibration blank must be less than the RL.	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Interference check standards (ICSA/ICSAB)	At the beginning of an analysis and after every 12 hours of analysis	Pay attention to false positives and false negatives for elements not present in the solutions. Results of the spiked elements should fall within +/- 20%.	If an element is consistently out of the +/- 20% range in the ICSAB solution, the problem should be investigated.
Serial Dilution (1/5 dilution)	One per batch of twenty samples or less	See Section 9.2.6	
Reporting Limit Check Standard (CRI)	At the beginning of analysis run	For *non-drinking water* analysis: Recovery +/- 50% of the true concentration. For *drinking water* analysis: Recovery +/- 50% of the true concentration for Sb, Ba, Be, Cd, Cr, Ni, Tl. All others +/- 15% of the true concentration	Stop the analysis, fix the problem and reanalyze the affected samples
Internal Standards	Analyzed with every standard and sample	200.8- 60-125%	Standard-terminate the analysis and recalibrate Sample- dilute (The analyst should check the IS response for the bracketing CCV/CCBs to see if there is a similar response to the sample.

QC Item	Frequency	Criteria	Corrective Action
			Recalibration may be required.)

9.2.1. Initial Calibration Verification (ICV): Analyze an initial calibration verification solution at the beginning of the run. ICV solution must have the same acid matrix as the calibration standard and it must come from a source other than the calibration standard source. The final concentrations of the various elements are listed in Attachment 1. The results for the target elements in the initial calibration verification (ICV) must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem and recalibrate the instrument. See Sec 7.2.4 for the ICV standard preparation instructions.

9.2.2. Continuing Calibration Verification (CCV): The calibration of the ICP-MS must be verified every 10 samples and at the end of the analysis run by analyzing the QC Check Solutions (CCV). For non-drinking waters, use Cal4/CCV. For drinking waters, use Cal3/CCV. The concentration of the CCV standard must be at or near mid-range levels of the calibration. The final concentrations of the various elements are listed in Attachment 1. The results for the target compounds must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the samples following the last good CCV.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB): ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. Use reagent water for the blank solution.

9.2.4. Reporting Limit Check Standard (CRI): At the beginning of the analysis run verify the accuracy at the RL by analyzing a solution at the RL. RL/PQL Check Solution is analyzed to demonstrate that the ICPMS is capable of detecting the target compounds at the reporting limit (RL). For non-drinking water analysis, the determined concentration must be within +/-50% of the true concentration. For drinking water analysis, the determined concentration must be within +/-50% of the true concentration for Sb, Ba, Be, Cd, Cr, Ni, and Tl; for all others, +/-15% of true concentration. See Attachment 1 for list of Rep Lim standard concentration in ug/L. Refer to Sec 7.2.8 for the RL/PQL standard preparation instructions.

9.2.5. Interference Check Sample (ICSA & ICSAB): Verify the inter-element and background corrections by analyzing the interference check solutions (ICSA & ICSAB) at the beginning of the analysis run and after every 12 hours of analysis. The ICS is analyzed in order to demonstrate that proper corrections are being utilized for known interferences. Analyst must evaluate these solutions in order to detect trends that require corrective actions. Pay particular attention to false positives and false negatives for

elements not present in the interference check solutions. Results should be within 20% of the true value for each element. See Attachment 2 for list of elements and the corresponding concentrations in ppb.

9.2.6. Serial Dilution QC Check: A 1/5 dilution is prepared and analyzed on one sample per batch (one for every ten samples for Method 200.8, or whichever is greater) to determine if matrix interferences are present.

9.2.6.1. Select a sample digestate that contains one or more target analytes at concentrations greater than 10X the reporting limit.

9.2.6.2. Dilute the digestate by a factor of 5 (DF=5) and analyze the dilution using the same procedures used for the un-diluted aliquot.

9.2.6.3. Compare the results of the diluted and un-diluted aliquots of sample digestate.

9.2.6.4. If the results of the dilution are within ±10% of the results of the undiluted sample, no matrix interference is present. If not, matrix interference should be suspected and the sample digestate should be subjected to a post-digestion spike.

9.2.7. Determination of Linear Range of the ICP-MS

Determine the linear dynamic range of the instrument by analyzing a minimum of three high standards at the upper limit of the instrument. The analytically determined concentration of these standards must be within 10% of the true value. The linear ranges should be redetermined annually or if the instrument is significantly changed.

9.2.7.1. Prepare the standard at concentrations that are expected to define the linear range of the instrument. The calibration standards and the linear range standards should be matrix matched; that is, they have the same percentage of hydrochloric and nitric acids. See Attachment 1 for Linear Range concentrations in ug/L.

9.2.7.2. Analyze the standard(s) after the initial calibration is validated.

9.2.7.3. Compare the concentration of the linear range standard with its true concentration using the following equation:

$$\text{Percent Difference} = \left|\frac{Ccal - Ctrue}{Ctrue}\right| \otimes 100$$

Where:

Ccal = concentration determined from analysis
Ctrue = true concentration of the standard

Note: If the percent difference is less than or equal to 10%, the linear range is confirmed at that concentration. If the percent difference is greater than 10%, repeat the analysis with a lower concentration. For elements validated in this manner you may report data up to 90% of that linear range before a dilution is required.

10.0. Procedure

10.1. Sample Preparation

10.1.1. For non-potable samples to be analyzed for total analytes by Method 200.8, follow TestAmerica SOP No. ED-MTP-001.

10.1.2. For Drinking water samples, if turbidity is ≥1, follow the digestion procedure for Total recoverable in TestAmerica SOP No. ED-MTP-001.

10.2. Calibration

10.2.1. Turn the ICP-MS on and initiate the tune screen. Start the tune screen to allow the instrument to become thermally stable before beginning to analyze the calibration standards. While the instrument is warming up, aspirate the interference check solution (or similar solution) to precondition the cones.

10.2.2. Check the tune parameters for the proper sensitivities, precision, oxides, and double charged values following the instrument manufacturer's recommendations. Refer to sections *Preparations Before Analysis*, *Plasma ON/Executing a Startup*, and *Appendix (Recommended Values for Tuning Parameters)* of the MassHunter Workstation User Guide (*Agilent Technologies 7800/7900, p/n G7201-90403).*

10.2.3. Prior to calibration, run an EPA Tune check solution. Refer to Sec. 7.2.7 for the Tune check criteria.

10.2.4. The instrument is required to be calibrated daily or once every 24 hours. For Non-Drinking Water analysis, use instrument method *BM New Method.icpms.template.* For Drinking Water analysis, use instrument method *DW New Method.icpms.template*. These are programs in the Mass Hunter Workstation software containing calibration standards, check tables, etc. Calibrate the instrument using a blank and five standards. Refer to the section *Creating a Method* in the instrument manufacturer's instructions *Agilent ICP-MS (7800/7900) MassHunter Workstation User Guide (p/n G7201-90403)*.

10.2.5. Internal standards are added to the calibration standards, blank and samples in-stream by the use of T-fitting.

10.2.6. Analyze the calibration standards and calibrate the ICP-MS in accordance with the manufacturer's recommendations and the TestAmerica Corporate

SOP No. CA-Q-S-005 Calibration Curves (General).

10.2.7. Calibration Curve Read-Back:

10.2.7.1. Low-Level readback – evaluate Cal 1 which is at the LLOQ. The %RE (relative error) must be +/- 50% (see Sec 11.4); if not, stop the analysis and recalibrate.

10.2.7.2. Mid-Level readback – evaluate Cal 4. The %RE (relative error) must be +/- 10% (see Sec 11.4); if not, stop the analysis and recalibrate.

10.3. Sample Analysis

10.3.1. All sample measurements must be made within 90% of the linear range of the instrument. Preparation of all reference materials used for calibration must be documented.

10.3.2. All potable and non-potable samples are typically analyzed undiluted. Dilutions must be prepared in reagent water (Sec 7.1.6 for Potable; Sec 7.1.7 for Non-Potable).

10.3.3. The samples are analyzed only after the ICB/CCB and ICV/CCV criteria are met.

10.3.4. The samples are analyzed in a sequence as follows:

Typical Analytical Sequence
INSTRUMENT WARM-UP (approx. 30 minutes)
TUNE CHECK
STANDARDIZATION/CALIBRATION (Blank, 5 Cal Stds)
ICV
ICB
CRI
ICSA
ICSAB
LRC-A (6020B)
LRC-B (6020B)
Rn Chk
Rn Chk
CCV
CCB
10 Samples
CCV
CCB
10 Samples
CCV
CCB
{continue until done; after 12 hrs run ICSA/ICSAB}

Note: The analytical sequence must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.3.5. Determine the concentration of the samples and QC items using the procedures of Section 11.

10.3.5.1. If the sample concentration is greater than 90% of the linear range, the sample digestate must be diluted and reanalyzed.

10.3.5.2. The amount of sample digestate needed to prepare the desired dilution is determined from the following equation:

$$\text{V digest} = \frac{Vf\ vol}{DF}$$

Where:
Vf vol = final volume of diluted sample (mL)
V digest = volume of sample digestate used to make the dilution (mL)
DF = Dilution Factor

10.3.5.3. The dilution factor is calculated as follows:

$$\text{DF} = \frac{Vf\ vol}{V\ digest}$$

Where: Vf vol = final volume of diluted sample extract (mL)
V digest = volume of sample extract used to make the dilution (mL)

Note: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE

A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP-MS. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with reagent water. What is the dilution factor?

$$\text{DF} = \frac{100ml}{1.0ml} = 100$$

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation:

$$\text{Concentration = ug/ L} = \frac{C \times V1 \times D}{V2}$$

Where:

C= Element concentration from instrument (ug/L)

V1= Final volume of sample digested (in liters)

D= Dilution performed on sample)

V2= Initial volume of sample digested (in liters).

11.4. Relative Error (%RE):

$$\text{\% Relative Error} = (100)\,(MC-TC)/TC$$

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Reduction

11.5.1. All data is recorded directly into the TALS Analyst Desktop II program.

11.5.2. Standard and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers.

11.5.3. Generate Raw Data PDF file for TALS

11.5.3.1. Using a scanner, scan all raw data, i.e., instrument printouts. This includes, the calibration, calibration curve info, and all samples analyzed (includes instrument and batch QC). Attach the pdf to the Docs section of the analytical batch in TALS (ADII).

11.5.4. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch.

11.5.4.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.
11.5.4.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'
11.5.4.3. Acknowledge by filling in responses to all unacknowledged findings.
11.5.4.4. Fill in appropriate comments in the response box, then hit 'OK.'
11.5.4.5. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

The IDL for EPA Method 200.8 is defined as the concentration equivalent to the analyte signal which is equal to 3 times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the selected mass(es) and should be determined annually.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out.

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization. The neutralization system consists of a 55-gallon poly open top container. There is also a local snorkel exhaust for venting any noxious odors. Once the liquid is transferred into the container, the contents are sparged with air and a representative pH is measured and the amount of material is recorded in the discharge log. The pH value is recorded in the discharge log, and the liquid is neutralized to a pH of 6-9 SU using food grade sodium bicarbonate. Once the secondary pH has been taken, confirmed and recorded, the discharge valve is opened to the municipal sewer system and the neutralization process is complete. The date, type of waste stream, and volume discharged are recorded on a discharge log.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  TSDF under Veolia Profile Number: 590598

- Soil Retain Samples - These containers are stored in the main sample-receiving laboratory cooler until they are designated for disposal. Once designated for disposal, the samples are transferred to the waste disposal area and placed into cubic-yard disposal containers, e.g., Jupiter-boxes. This waste is shipped directly to the TSDF under Veolia Profile Number 402535 and is subject to incineration.

15.0. References / Cross-References

15.1. Methods for the Determination of Metals in Environmental Samples; US EPA Office of Research and Development. Washington, DC, May 1994. Method 200.8, Revision 5.4.

15.2. TestAmerica Corporate SOP No. CA-Q-S-005, *Calibration Curves (General)*, most current revision.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.6. TestAmerica Edison SOP ED-MTP-001, *Digestion of Aqueous Samples for Analysis by ICP and ICPMS USEPA Method No(s). 200.2, 200.7 (Rev 4.4), 200.8 & CLP SOW ISM01.2*, most current revision.

15.7. Agilent ICP-MS (7800/7900) MassHunter Workstation User Guide, p/n G7201-90403, Rev. A, June 2017

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Standards and Matrix Spiking concentrations – Drinking water and non Drinking water.
Attachment 2: Interference Check standard elemental concentrations - Drinking water and non Drinking water.

18.0. Revision History

- **Revision 14, dated 15 January 2023**
  - Sec 1.1, Table1: Updated reporting limit for Al and Zn in Drinking water, and Ca & Na for NPW to reflect current laboratory limits.
  - Sec 2.0 & 8.0: Clarified instructions for the analysis of dissolved samples (i.e., digestion not required for the analysis of dissolved samples).
  - Sec 7.2.2: Updated the preparation instructions for the Working calibration standards (drinking water and non-drinking water) to reflect current lab practices.
  - Sec 7.2.4.2.2: Updated the preparation instructions for the ICV Non-Potable.
  - Sec 7.2.4.2.2: Added preparation instructions for Interm_ICV std
  - Sec 7.2.6: Replaced CAL3/CCV with CAL4/CCV.
  - Sec 7.2.7.2: Updated preparation instructions for the potable Working Internal standard; Added titanium to the list of analytes in the no-gas mode; Updated

concentration of tune check analytes.
- Sec 7.2.7.4: Updated preparation instructions for the Working EPA Tune Check Std.
- Sec 9.2, Table 2: Revised the minimum number of standards to 5 and 1 blank.
- Sec 10.1: Revised to specify 'total analytes' for samples which require digestion prior to analysis.
- Sec 14.2: Updated waste management information to reflect current practices.
- Attachment 1, Table for Drinking water standards: Updated Cal-3 standard concentrations to reflect current lab practices.

- **Revision 13, dated 08 October 2020**
  - Sec 1.1, Table1: Updated the Drinking water reporting limit concentrations for Sb, Ba, Be, Cd, Cr, Ni, Se, and Tl to reflect current laboratory limits.
  - Sec 3: Updated the Definitions locations as referenced in the Laboratory Quality Manual.
  - Sec 7.1.6: Updated the preparation instructions for the reagent water.
  - Sec 7.2: Added instructions to make varying amounts of standards
  - Sec 7.2.1: Removed stock standards TANJ-STD-1, TANJ-STD-2, TA-88, STLNJ-STD-1, STLNJ-STD-2, and TA-72REV2 from this SOP; Added stock standard 200.8 Potable RL standards.
  - Sec 7.2.2: Updated preparation instructions for Drinking Water standards.
  - Sec 7.2.3: Deleted stock standard ICV1ICPMS and ICV2ICPMS, no longer applicable.
  - Sec 7.2.4.1: Updated preparation instructions for ICV (Potable).
  - Sec 7.2.7.2: Added preparation instructions for the Potable working internal standard.
  - Sec 7.2.9: Deleted Interference stock standards 6020ICS-8A & 6020ICS-8B.
  - Sec 7.2.10: Updated preparation instructions for ICSA/ICSAB drinking water.
  - Sec 9.1.6.2: Updated Non-Potable PDS spiking instructions; updated Non-Potable PDS concentrations in Table.
  - Sec 9.2, Table 2 & Sec 9.2.4:Updated reporting limit (CRI) criteria limits for Sb, Ba, Be, Cd, Cr, Ni, Tl.
  - Sec 18.0, Attachment 1 (Potable): Updated calibration, ICV, CCV concentrations.
  - Sec 18.0, Attachment 2 (Potable): Updated ICSA & ICSAB concentrations

- **Revision 12, dated 18 June 2020**
  - Updated SOP header to Eurofins emblem.
  - Throughout the SOP removed all SW846 Method 6020 references.
  - For Non-Drinking Water preparations: Throughout the SOP, replaced all references to 2%HNO3/0.5%HCl with 5%HNO3/5%HCl.
  - Sec 1.1, Table 1: Removed references to typical dilutions and updated the RLs for Non-Drinking Water.
  - Sec 6.1: Added two ICPMS instruments, Agilent 7800 & 7900; removed reference to resolution and grating; added instrument mass range. Updated the models of the heat exchanger and autosampler.
  - Sec 7.1.7: Added preparation instructions for 5%HNO3/5%HCl.
  - Sec 7.2.1: Added CPI stock standard concentrations; CPI International is added as the vendor for the stock cal stds.

- Sec 7.2.2: Updated preparation instructions for the non-drinking water working cal standards and added preparation instructions for MS-Cal-Int.
- Sec 7.2.3: Updated the Initial calibration verification stock standards.
- Sec 7.2.4.2: Updated preparation instructions for the non-potable ICV working standard.
- Sec 7.2.4.2.1: Added preparation instructions for MS-ICV-Int.
- Sec 7.2.7.1.2: Replaced Inorganic Ventures with CPI International as the vendor source for the Ge stock standard.
- Sec 7.2.7.2: Updated preparation instructions for the Internal standard. Changed recommended mass for Ca (non-potable) from 44 to 40. Updated typical referenced internal std for As & Se from Ge to Sc. Updated typical analysis mode for non-potable Ca & Fe. Added Ca44 as the typical mass used for Calcium during potable analysis.
- Sec 7.2.9 & 7.2.10: Added ICSA/ICSAB stock standards from CPI International and updated Non-Potable preparation instructions for working ICSA & ICSAB.
- Sec 9.1 & 9.11: Updated control limit criteria for the Preparation blank.
- Sec 9.1.2 & 9.1.5: Updated preparation instructions for the LCS water samples and aqueous matrix spikes (MS).
- Sec 10.2.2: Added reference material, MassHunter Workstation User Guide, for the ICPMS instruments 7800 & 7900. Removed references to ICPMS1&2 (includes Chemstation software and relating Operation Manuals) from this SOP.
- Sec 10.2.4: Added instrument method program and reference material, MassHunter Workstation User Guide, for the ICPMS instruments 7800 & 7900.
- Sec 10.2.7.1 & 10.2.7.2: Added Low-Level readback and Mid-Level readback evaluation instructions.
- Sec 10.3.2: Updated typical dilution instructions.
- Sec 10.3.4: Updated Typical Analytical Sequence table to reflect current laboratory practices.
- Sec 11.3.2: Added final results calculation instructions.
- Sec 11.5: Added %Relative Error calculation equation.
- Sec 11.6.5: Added instructions for the Data Review Checker (DRC); removed reference to the Metals data review checklist EDS-WI-146, not applicable.
- Sec 15.3, 15.11, & 15.12: Deleted references for the Agilent 7500 Manuals, not applicable.
- Sec 15.13: Deleted reference to the ICPMS Data Review Checklist, CA-Q-WI-044.
- Sec 15.14: Added reference for the Agilent ICPMS 7800/7900 User Guide.
- Sec 18.0, Attachment 1 (Non-Potable): Updated calibration, ICV, CCV concentrations; removed dilution instructions.
- Sec 18.0, Attachment 2 (Non-Potable & Potable): Updated ICSA & ICSAB analyte list and concentrations.

- **Revision 11, dated 26 April 2018**
  - Sec 7.2.7.4: Added Magnesium as one of the working tune check solution's constituents.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS in soil matrix.

- **Revision 10, dated 15 November 2016**

- Sec 1.1, Table 1: Changed the Drinking water RL from 2.5 ug/L to 2 ug/L. Removed reference to Method 3005.
- Sec 2.0: Removed reference to method 3005 and replaced SOP ED-MTP-007 with SOP ED-MTP-001.
- Sec 7.1.6: Reagent recording in ICPMS Reagent Dilution logbook is replaced with TALS Reagent Module.
- Sec 7.2.1: Replaced stock standard TA-72 with standard TA-72REV2.
- Sec 7.2.2: Replaced standard TA-72 with TA-72REV2 for CAL1 standard-Drinking Water.
- Sec 7.2.7.2: Tune check criteria: updated the resolution check criteria to reflect laboratory practices.
- Sec 7.2.10: Added the preparation instructions for ICSA & ICSAB standards used when analyzing drinking water samples.
- Sec 9.2, Table 2: Updated Tune validation-Resolution Check criteria to reflect laboraotry practices
- Sec 10.1: Replaced SOP ED-MTP-007 reference with SOP ED-MTP-001.
- Sec 11.5.4: Replaced checklist CA-Q-WI-004 with Work Instruction EDS-WI-146.
- Attachment 1, Drinking water: Replaced the Calibration concentration and RL Std.Conc. of Pb from 2.5 to 2 ug/L.
- Attachment 2: Added ICSA & ICSAB elemental concentrations for Drinking water analysis.

- **Revision 9, dated 02 April 2015**

  - Throughout the SOP: Removed all reference to SOP# ED-MTP-002.
  - Sec 7.2.1: Added calibration standards TA-88 and TA-72.
  - Sec 7.2.2: Added the new low-level cal std (CAL2) for non-potable water and updated the preparation instructions for the potable cal standards.
  - Sec 7.2.7.1.3: Added Triton X-100 to the list of internal standard stock solutions.
  - Sec 7.2.7.2: Updated the preparation instruction of the working internal standard solution to include Triton X-100; Revised note to state that Selenium is now analyzed under the Hydrogen reaction mode.
  - Sec 7.2.8: CAL1 is now used as the RL check standard for drinking water analysis.
  - Sec 9.1.6.4: Added PDS recovery limits (85-115%) for method 200.8. Clarified the procedure when PDS is outside the acceptable limits.
  - Sec 9.1 and 9.1.7: Added Matrix Spike Duplicate (MSD) to the list of Sample QC.
  - Sec 11.5.4: Updated the metals data review checklist form number.
  - Sec 15.13: Added TestAmerica Corp form No. CA-Q-WI-044 to the list of references.
  - Attachment 1 (non-Drinking Waters): Updated the calibration concentrations for all metals; updated the matrix spike concentration for Zinc (water & soil).
  - Attachment 1 (Drinking Waters): Updated the calibration concentrations for As, B,Sn and Zn.

- **Revision 8, dated 05 December 2012**

  - Sec 1 and 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 2.0: Deleted the exception that silver analysis does not undergo digestion. Silver may be directly analyzed for drinking waters provided the turbidity <1.
  - Sec 7.1.6: Revised to include the reagent water prep for potable samples not

requiring digestion. Revised preparation frequency to 12 months to reflect actual laboratory practices.
- Sec 7.2.2 & 7.2.3: Sections deleted to remove Hg stock standard info and the preparation instructions of Hg intermediate standard; subsequent sections adjusted accordingly.
- Sec 7.2.2 (formerly Sec 7.2.4): Removed all references to Hg. Clarified M1CAL3 as the reporting limit for Drinking Water standard. Clarified M1CAL2 is not the reporting limit for Boron.
- Sec 7.2.3 and 7.2.4 (formerly Sec 7.2.5 & 7.2.6): Hg references have been removed.
- Sec 7.2.6: Section added; subsequent sections adjusted accordingly.
- Sec 7.2.7 (formerly Sec 7.2.8): Added 'EPA Tune Check' to the section header. Removed the use of gold standard from this SOP. Hg is not analyzed by this method.
- Sec 7.2.7.1. Added internal standard stock solutions.
- Sec 7.2.7.2: Added internal standard preparation instructions. Also clarified that drinking water samples are analyzed without the use of collision cell technology.
- Sec 7.2.7.3: Added Tune Check stock standard
- Sec 7.2.7.4: Added preparation instructions of the EPA Tune Check standard.
- Sec 7.2.11 (formerly Sec 7.2.12): Changed standard vendor for the Matrix spike stock solution to High-Purity Standards.
- Sec 8: Revised the wait time for sample preparation after in-lab preservation to 24 hours to comply with the updated 40CFR Part 136.
- Sec 9.1: Preparation Blank's control limits have been updated to reflect method requirements.
- Sec 9 and 10: Revised section references of standard solutions where applicable.
- Sec 10.1.2: Revised SOP number reference to reflect actual laboratory practices.
- Sec 10.2.2: Included Tuning Handbook reference for ICPMS2.
- Sec 10.2.4: Included Operation Manual for Masshunter software to ICPMS2.
- Sec 11.5: Added instructions for data recording, raw data PDF creations and Metals Data Review Checklist.
- Sec 15.4: Deleted SOP No. ED-MTP-007 reference, not applicable; subsequent sections adjusted accordingly.
- Sec 15.11 through 15.13: Added applicable references.
- Sec 17.0: Attachments 3 and 4 have been removed.
- Attachment 1: Deleted 'Linear range conc.' column; new linear range studies are updated on instruments. Updated MS concentration for Boron.

- **Revision 7, dated 16 July 2010**

  - Sec 1.1 Table 1: Revised Reporting Limits for drinking water and Soil.
  - Sec 3: Revised definitions reference to Appendix 2 of ED-QA-LQM.
  - Sec 7.2.1: Added STLNJ-STD-1 and STLNJ-STD-2 to the list of Calibration Stds
  - Sec 7.2.4: Revised working calibration standard including preparation procedure to reflect actual lab practices.
  - Sec 7.2.9: Revised prep procedure for RL check standard. Also deleted RL/PQL working standard, previously section 7.2.10. Subsequent sections adjusted accordingly.
  - Sec 18.0 Attachment 1: Table updated to reflect correct calibration standard concentrations and drinking water reporting limits.

- **Revision 6, dated 11 September 2009**

- Sec 9.2 Table 2 & Sec. 9.2.4: Revised Reporting limit criteria for drinking water analysis to ± 15%.

- **Revision 5, dated 08 July 2009**

  - Sec 4.1: Expanded section to include the isobaric polyatomic/molecular ion interferences; clarified using collision/reaction cell for non-potable samples only.
  - Sec 4.2: Added equations used for drinking water analysis when collision/reaction cell can not be used.
  - Sec 4.3: Added Krypton interference which affects arsenic and selenium, and the use of high purity Krypton free argon.
  - Sec 6.1 Instrumentation: Add collision/reaction cell.
  - Sec 7.2.4: Added a low calibration standard (M1CAL1) increasing the number of calibration standards to five; five cal standards will only be analyzed for elements Be, Fe, Pb and Mn.
  - Sec 7.2.8: Added Kr83 in the list of elements in order to monitor Kr levels.
  - Added text in Tune check criteria, Sec 7.2.8: *Resolution is checked by analyzing Magnesium isotopes 24, 25, 26 and Lead isotopes 206, 207, 208.*
  - Sec 8: Clarified holding time and procedure for acidifying unpreserved samples.
  - Sec 9: Added text: *If a batch of samples requires digestion, then the relating QC samples must be carried through the entire digestion process.*
  - Sec 9.1 & 9.1.2: Changed LCS control limit for Method 6020 from ± 25% to ± 20%.
  - Sec 9.2.2: CCV standard- changed solution to M1CAL4.
  - Sec 10.2.4: Calibration- changed the number of standards from three to four standards.
  - Sec 10.3.2: Added in the procedure the requirement for diluting samples 5x (water) and 20x (soil) prior to analysis; subsequent section numbers were adjusted accordingly.
  - Sec 10.3.5.1: revised to include that samples will be diluted if the concentration is >90% of the linear range.
  - Attachment 1: Revised calibration standard concentration specifically Cal std 2 to reflect the correct concentration. Additionally, a fifth cal standard was added to the four elements (Be, Fe, Pb, and Mn) so the RL will be at or above the low level standard; Reporting limits were also revised for these elements.
  - Attachment 3: Revised dilution requirement for soil analysis from 10x to 20x.

- **Revision 4, dated 24 March 2009**
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1: Relocated Table 1 to this section. Updated 6020 Soil RLs. Revised several RLs for Drinking Water and Digested Water
  - Sec. 2: Added the method and SOP references for digestion of samples to be analyzed for metals via ICPMS.
  - Sec 6.1: Added Heat exchanger and autosampler to the list of instruments.
  - Sec 7.1.6: Added reagent water and its prep instructions to the list of reagents.
  - Sec 7.2 Standards: Included vendor and storage information of standards.
  - Sec 7.2.2: Revised the vendor source and stock concentration of the Hg standard from 1000 ppm to 10 ppm; revised preparation instructions of the Intermediate standard (Hg-Cal-Int).

- Sec 7.2.4: Revised prep instructions for the working calibration standards (i.e. amount of spiking solution and final volume).
- Sec 7.2.5: Deleted ICV stock standards STLNJ-Cal 6 & 7 and added ICV stock standards ICVICPMS and ICV2ICPMS.
- Hg Secondary stock solution in Sec 7.2.5: Revised the vendor source and stock concentration of the Hg secondary standard from 1000 ppm to 10 ppm; revised preparation instructions of the Intermediate standard (Hg-ICV-Int).
- Sec. 7.2.8: added statement: "Drinking Water Analysis: All tune steps = 3 (thereby eliminating the use of the Collision/Reaction Cell.) Use masses Fe57, Cu65, and Se82 instead of their respective masses listed above."
- Sec 7.2.9: Deleted ICPMS RL stock standard and added custom standards (STLNJ-STD-1 & 2) for Reporting Limit Check standard.
- Sec 7.2.10: Revised prep instructions for the RL/PQL Working standard.
- Sec 7.2.11: Revised prep instructions for the Working Interference check standards (final volume reduced from 100ml to 50ml; spiking amount adjusted).
- Sec 8: Sample collection, preservation and storage: Reformat section into Table format and included minimum sample size and reference information.
- Sec. 9.1: Added prep instruction to the QC samples (MB, LCS, BS and MS).
- Deleted Single point method of standard addition; previously identified Sec.9.2.8.
- Sec 10.1: Clarified the digestion procedure utilized for digesting different matrices (wastewater, drinking water and soil).
- Sec 15: Added applicable references.
- Sec 17: Added Attachment 2 (Interference Check Standard elemental concentrations).

- **Revision 3, dated March 2007**
  - Section 16. Attachment 1. Modified the 'Linear Range Concentration' of 11 elements (Sb, Be, B, Cd, Co, Pb, Se, Ag, Sr, Tl, V) to reflect the most recent LDR study.

- **Revision 2**, **dated February 2007**
  - Revised various sections of the SOP to include the specific sections in the Operator's manual where applicable information can be found. These sections are Sec. 5.3, 10.1.2., 10.1.3, Appendix A – Analytical Sequence Table.
  - Section 6.3 Calibration Standards. Modified the frequency for the preparation of calibration standards from six months to two weeks as per Method 200.8.
  - Section 6.4. Linearity Check Standards. Revised the HNO3 acid concentration used in the preparation of linearity check solutions to '2% by volume' to reflect actual laboratory practices.
  - Section 6.5 (Internal Standards). Revised the concentration of the element Au, both spiking and resulting concentrations.

    '*A solution of 1 mg/L (internal standard elements) and 2 mg/L of Au is prepared and added in-stream by the use of a T-fitting. The resulting concentration (in-stream) of the standard is ~100 ug/L (Au) and ~50 ug/L (internal standard elements).'*

  - Section 7.4. Section deleted; contents not applicable to this SOP.
  - Section 8.1.1 (MDL and IDL determination). Modified the solution used in the analysis of IDL to 'reagent blank solution' to reflect actual laboratory practices.
  - Section 8.1.2 (MDL and IDL determination). Added an SOP reference to further describe MDL determination.

- Section 10.3.2. The name of the Reporting limit solution in the analysis sequence was changed from 'Detection Limit Check Solution' to 'Reporting Limit Standard (RL).'
- Section 15.1, deleted; reference not applicable. Subsequent section numbers adjusted accordingly.
- Section 15.1 (previously identified as section 15.2), was revised to include the specific method in the reference information, in this case method 6020.
- Section 15.2 (previously identified as section 15.3), was revised to include the specific method in the reference information, in this case method 200.8.
- Section 15.6. Added Operator's instructions manual in the list of references. The section previously identified as 15.6 is now 15.5.
- Section 16. Attachment 1. Modified the 'Linear Range Conc. (ug/L)' to reflect the most recent LDR study. Updated the 'RL Std. conc.' for Boron, Iron, Tin and Zinc to reflect actual laboratory's reporting limits for these elements.
- Appendix A (Sample Preparation). Specified the name of the SOP used in the digestion of samples.
- Appendix A (Calibration, Quality Control and Corrective action summary). Revised the criteria for the ICSA/ICSAB to clarify that ICSA when used as linear range sample the determined analyte concentrations >90% must be diluted and reanalyzed.
- Appendix B (ICV Solution). Added the frequency for the preparation of the ICV solution.

- **Revision 1, dated January 2007**

  - Specified Operator's manual '*Agilent 7500 ICP-MS Chemstation (G1834B)*' in various sections: Sections 5.3, 10.1.2, 10.1.3, Appendix A – Analytical Sequence section.
  - All references to '6020A' are updated to '6020' to reflect actual method reference (i.e. sections 1.1, 2.2, 8.1.2 etc.).
  - Section 10.1.3. Filename '2008.M' is specified as the program/filename used in the ChemStation software.
  - Section 16 Table 1. Updated Reporting limits of 8 elements (As, Cd, Co, Cu, Ni, Ag, Sr, Sn) to reflect current standard concentrations and laboratory's reporting limits.
  - Section 16 Attachment 1. Revised Matrix spike concentrations for water and soil to include the actual final expected concentrations of the matrix spike.
  - Appendix B – Calibration, Quality Control and Corrective action summary. Criteria for internal standards was changed from >30% to 30-120% to reflect actual method criteria limits.
  - Appendix B – ICP Interference Check Solutions for SW846 6020. Added the frequency of preparation for the Working Interference Standards ICSA and ICSAB, 'these standards are made fresh weekly.'
  - Appendix B. Added data qualifiers for ICP-MS
  - Section 15.4. Added USEPA CLP ILM05.3 to the list of references.

Attachment 1

STANDARDS AND MATRIX SPIKING CONCENTRATIONS
For Non-Drinking Waters

Name	Mass	Cal1 CRI	Cal-2	Cal-3	Cal-4/CCV	Cal-5	ICV	*LCS/MS Conc*
		(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	*(ug/L)*
Be	9	0.8	4	50	100	200	80	*25*
B	11	80	400	125	250	500	200	*500*
Na	23	200	1000	12500	25000	50000	20000	*2500*
Mg	24	200	1000	12500	25000	50000	20000	*2500*
Al	27	40	200	12500	25000	50000	20000	*2500*
K	39	200	1000	12500	25000	50000	20000	*2500*
Ca	40	200	1000	12500	25000	50000	20000	*2500*
Ti	47	4	20	50	100	200	80	*50*
V	51	4	20	50	100	200	80	*50*
Cr	52	4	20	50	100	200	80	*50*
Mn	55	8	40	50	100	200	80	*250*
Fe	56	120	600	12500	25000	50000	20000	*2500*
Co	59	4	20	50	100	200	80	*25*
Ni	60	4	20	50	100	200	80	*50*
Cu	63	4	20	50	100	200	80	*50*
Zn	66	16	80	125	250	500	200	*250*
As	75	2	10	50	100	200	80	*50*
Se	78	2.5	12.5	50	100	200	80	*50*
Sr	88	4	20	50	100	200	80	*50*
Mo	95	4	20	50	100	200	80	*50*
Ag	107	2	10	25	50	100	40	*25*
Cd	111	2	10	50	100	200	80	*25*
Sn	118	16	80	50	100	200	80	*50*
Sb	121	2	10	50	100	200	80	*25*
Ba	137	4	20	50	100	200	80	*50*
Tl	205	0.8	4	50	100	200	80	*20*
Pb	208	1.2	6	50	100	200	80	*25*

STANDARDS AND MATRIX SPIKING CONCENTRATIONS
For Drinking Waters

Name	Mass	Cal1 CRI	Cal-2	Cal-3	Cal-4/CCV	ICV	*LCS/MS Conc*
		(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	*(ug/L)*
Be	9	0.3	10	25	100	40	*5*
B	11	20	20	50	200	80	*100*
Na	23	250	500	1250	5000	2000	*500*
Mg	24	250	500	1250	5000	2000	*500*
Al	27	25	500	1250	5000	2000	*500*
K	39	250	500	1250	5000	2000	*500*
Ca	44	250	500	1250	5000	2000	*500*
Ti	47	2.5	10	25	100	40	*10*
V	51	2.5	10	25	100	40	*10*
Cr	52	0.9	10	25	100	40	*10*
Mn	55	25	10	25	100	40	*50*
Fe	57	250	500	1250	5000	2000	*500*
Co	59	2.5	10	25	100	40	*5*
Ni	60	0.5	10	25	100	40	*10*
Cu	65	2.5	10	25	100	40	*10*
Zn	66	4	10	25	100	40	*50*
As	75	1	10	25	100	40	*10*
Se	82	8	10	25	100	40	*10*
Sr	88	2.5	10	25	100	40	*10*
Mo	95	2.5	10	25	100	40	*10*
Ag	107	2.5	10	25	100	40	*5*
Cd	111	0.5	10	25	100	40	*5*
Sn	118	4	10	25	100	40	*10*
Sb	121	0.4	10	25	100	40	*5*
Ba	137	2	10	25	100	40	*10*
Tl	205	0.3	10	25	100	40	*4*
Pb	208	2	10	25	100	40	*5*

Attachment 2

Interference Check Standard (ICSA & ICSAB) Elemental Concentrations				
ELEMENT	Non-Potable		Potable	
	ICSA (ug/L)	ICSAB (ug/L)	ICSA (ug/L)	ICSAB (ug/L)
Sodium	100000	100000	25000	25000
Magnesium	100000	100000	25000	25000
Aluminum	100000	100000	25000	25000
Potassium	100000	100000	25000	25000
Calcium	100000	100000	25000	25000
Titanium	2000	2000	500	500
Iron	100000	100000	25000	25000
Molybdenum	2000	2000	500	500
Carbon	200000	200000	50000	50000
Chloride	1000000	1000000	250000	250000
Phosphorus	100000	100000	25000	25000
Sulfur	100000	100000	25000	25000
Silver	n/a	200	n/a	50
Arsenic	n/a	200	n/a	50
Barium	n/a	200	n/a	50
Beryllium	n/a	200	n/a	50
Cadmium	n/a	200	n/a	50
Cobalt	n/a	200	n/a	50
Chromium	n/a	200	n/a	50
Copper	n/a	200	n/a	50
Lithium	n/a	200	n/a	50
Manganese	n/a	200	n/a	50
Nickel	n/a	200	n/a	50
Lead	n/a	200	n/a	50
Antimony	n/a	100	n/a	25
Selenium	n/a	200	n/a	50
Silicon	n/a	200	n/a	50
Tin	n/a	200	n/a	50
Strontium	n/a	200	n/a	50
Thallium	n/a	100	n/a	25
Vanadium	n/a	200	n/a	50
Tungsten	n/a	2000	n/a	500
Zinc	n/a	200	n/a	50

Title: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW846 Methods 8270E

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

USEPA Method 8270E is an analytical method which employs the use of GC/MS to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples

Eurofins Edison has the capability to analyze and report the compounds listed in Table 1 via Method 8270E.

Table 1

Compound	CAS No.	Compound	CAS No.
1,1'-Biphenyl	92-52-4	Anthracene **(1)**	120-12-7
1,2,4,5-Tetrachlorobenzene	95-94-3	Atrazine	1912-24-9
1,2,4-Trichlorobenzene	120-82-1	Benzaldehyde	100-52-7
1,2-Dichlorobenzene	95-50-1	Benzidine	92-87-5
1,2-Diphenylhydrazine	122-66-7	Benzo[a]anthracene **(1)**	56-55-3
1,3-Dichlorobenzene	541-73-1	Benzo[a]pyrene **(1)**	50-32-8
1,3-Dimethylnaphthalene	575-41-7	Benzo[b]fluoranthene **(1)**	205-99-2
1,4-Dichlorobenzene	106-46-7	Benzo[g,h,i]perylene **(1)**	191-24-2
1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1	Benzo[k]fluoranthene **(1)**	207-08-9
1,4-Dioxane **(1) (2)**	123-91-1	Benzoic acid	65-85-0
1-Methylnaphthalene	90-12-0	Benzyl alcohol	100-51-6
1-Naphthylamine	134-32-7	Bis(2-chloroethoxy)methane	111-91-1
2,2'-oxybis[1-chloropropane]	108-60-1	Bis(2-chloroethyl)ether **(1)**	111-44-4
2,3,4,6-Tetrachlorophenol	58-90-2	Bis(2-ethylhexyl) phthalate	117-81-7
2,3,7,8-TCDD	1746-01-6	Bisphenol-A	80-05-7
2,3-Dihydroindene	496-11-7	Butyl benzyl phthalate	85-68-7
2,3-Dimethylaniline	87-59-2	Caprolactam	105-60-2
2,4,5-Trichlorophenol	95-95-4	Carbamazepine	298-46-4
2,4,5-Trimethylaniline	137-17-7	Carbazole	86-74-8
2,4,6-Tribromophenol **(Surrogate)**	118-79-6	Chrysene **(1)**	218-01-9
2,4,6-Trichlorophenol	88-06-2	Chrysene-d12 **(ISTD)**	1719-03-5
2,4-Dichlorophenol	120-83-2	Coumarin	91-64-5
2,4-Dimethylphenol	105-67-9	Dibenz(a,h)anthracene **(1)**	53-70-3
2,4-Dinitrophenol	51-28-5	Dibenzofuran	132-64-9
2,4-Dinitrotoluene	121-14-2	Diethyl phthalate	84-66-2
2,4-Xylidine	95-68-1	Dimethyl phthalate	131-11-3
2,6-Dinitrotoluene	606-20-2	Di-n-butyl phthalate	84-74-2
2-Chloronaphthalene	91-58-7	Di-n-octyl phthalate	117-84-0
2-Chlorophenol	95-57-8	Fluoranthene **(1)**	206-44-0
2-Ethylaniline	578-54-1	Fluorene **(1)**	86-73-7
2-Fluorobiphenyl **(Surrogate)**	321-60-8	Hexachlorobenzene **(1)**	118-74-1
2-Fluorophenol **(Surrogate)**	367-12-4	Hexachlorobutadiene	87-68-3
2-Methylnaphthalene	91-57-6	Hexachlorocyclopentadiene	77-47-4
2-Methylphenol	95-48-7	Hexachloroethane	67-72-1
2-Naphthylamine	91-59-8	Indeno[1,2,3-cd]pyrene **(1)**	193-39-5
2-Nitroaniline	88-74-4	Isophorone	78-59-1
2-Nitrophenol	88-75-5	n,n'-Dimethylaniline	121-69-7
2-tertbutyl-4-methylphenol	2409-55-4	Naphthalene **(1)**	91-20-3
2-Toluidine	95-53-4	Naphthalene-d8 **(ISTD)**	1146-65-2

Table 1

Compound	CAS No.	Compound	CAS No.
3 & 4 Methylphenol	15831-10-4	n-Decane	124-18-5
3,3'-Dichlorobenzidine	91-94-1	Nitrobenzene	98-95-3
3,4-Dimethylaniline	95-64-7	Nitrobenzene-d5 **(Surrogate)**	4165-60-0
3,5-di-tert-butyl-4-hydroxytol	128-37-0	N-Nitrosodimethylamine **(1)**	62-75-9
3-Nitroaniline	99-09-2	N-Nitrosodi-n-propylamine	621-64-7
4,6-Dinitro-2-methylphenol **(1)**	534-52-1	N-Nitrosodiphenylamine	86-30-6
4-Bromophenyl phenyl ether	101-55-3	n-Octadecane	593-45-3
4-chloro-2-methylaniline	95-69-2	o-Toluidine-d9 **(Surrogate)**	194423-47-7
4-Chloro-3-methylphenol	59-50-7	Pentachloronitrobenzene	82-68-8
4-Chloroaniline	106-47-8	Pentachlorophenol **(1)**	87-86-5
4-Chloroaniline–d4 **(Surrogate)**	191656-33-4	Perylene-d12 **(ISTD)**	1520-96-3
4-Chlorophenyl phenyl ether	7005-72-3	Phenanthrene **(1)**	85-01-8
4-Methylphenol	106-44-5	Phenanthrene-d10 **(ISTD**)	1517-22-2
4-Nitroaniline	100-01-6	Phenol	108-95-2
4-Nitrophenol	100-02-7	Phenol-d5 **(Surrogate)**	4165-62-2
Acenaphthene **(1)**	83-32-9	Phenyl ether	101-84-8
Acenaphthene-d10 **(ISTD)**	15067-26-2	Pyrene **(1)**	129-00-0
Acenaphthylene **(1)**	208-96-8	Pyridine	110-86-1
Acetophenone	98-86-2	Terphenyl-d14 **(Surrogate)**	1718-51-0
Aniline	62-53-3	Total Cresols	STL00160
Aniline-d5 **(Surrogate)**	4165-61-1		

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).
(2) Compound can also be analyzed by Isotope Dilution/SIM.

1.2 For a listing of method detection limits (MDLs) and Reporting Limits (RLs) please refer to the currently active Method 8270 Method Limit Groups in TALS (Eurofins LIMS).

1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work)*, and Section 19 (*Test Methods and Method Validation)* in Eurofins Edison's Quality Assurance Manual (Eurofins Edison Document No. ED-QA-LQM).

1.4 Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP ED-GEN-003. The NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

2.0 Summary of Method

2.1 This method is used for the analysis of aqueous and solid matrices for semi-volatile base, neutral and acid organic compounds that are extracted from the sample matrix with an organic solvent.

2.2 An aliquot of sample containing surrogate spiking compounds is extracted with an organic solvent. The extract is concentrated on a steam bath to a suitable volume. Internal standards are added to the extract.

2.3 Sample extraction techniques are specified for each matrix in the following Eurofins Edison SOPs:

- ED-ORP-002 (*Extraction of Semivolatile Organic Compounds in Water by Separatory Funnel, SW846 Method 3510C*);
- ED-ORP-043 (*SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270);*
- ED-ORP-0044 (*Microwave Extraction for Solids, SW846 Method 3546*);

2.4 A small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to a mass spectrometer (MS) which is used to detect the compounds eluting from the GC. The detected compounds are fragmented with an electron beam to produce a mass spectrum which is characteristic of the compound introduced into the MS. Identification of target analytes is accomplished by comparing their mass spectra with the electron ionization spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion (quantitation ion) relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8270E as applicable.

2.5 The standard preparation procedure for aqueous samples involves use of a Reduced Volume Extraction (250 ml) (RVE) followed by analysis using a Large Volume Injection (LVI). Optionally, a full volume (1000 ml nominal) may be employed. The details of the extractions are outlined in the applicable prep SOPs while the analytical details for 8270E is presented in this SOP.

2.6 These methods are also applicable to the analysis of samples by Selected Ion Monitoring (SIM) for the purpose of obtaining lower reporting limits for the following compounds:

Table 2 – SIM Analytes

SIM Analytes	CAS #
1,4-Dioxane	123-91-1
4,6-Dinitro-2-methylphenol	534-52-1
Acenaphthene	83-32-9

Table 2 – SIM Analytes	
SIM Analytes	**CAS #**
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzo[a]pyrene	50-32-8
Benzo[b]fluoranthene	205-99-2
Benzo[g,h,i]perylene	191-24-2
Benzo[k]fluoranthene	207-08-9
Bis(2-chloroethyl)ether	111-44-4
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Indeno[1,2,3-cd]pyrene	193-39-5
Naphthalene	91-20-3
N-Nitrosodimethylamine	62-75-9
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Pyrene	129-00-0

2.7 An isotope dilution selected ion monitoring (SIM) technique for the analysis of 1,4-dioxane in water at a reporting level of 0.2 ug/l is also described in this SOP. Using this technique 1,4-dioxane-d8 is added prior to sample extraction and is used as an internal standard to calculate the concentration of 1,4-dioxane present. Additionally, 1,4-dichorobenzene-d4 is added to the extract prior to analysis to monitor the recovery of 1,4-dioxane-d8.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of the Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Analysts must take steps to determine the source of the interference and take corrective action to eliminate the problem.

4.1.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce

carryover, the sample syringe is automatically rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross-contamination. Alternately, verify that the sample analyzed after the high concentration sample does not show any carryover through inspection of chromatogram and target results.

4.1.2 Contaminants from the extraction process detected in the method blank should be evaluated to determine the impact on the analysis. Interferences from any target analyte must not be present in the method blank above the reporting limit for that compound. If these types of interferences occur, corrective action is required. The source should be identified and corrective action initiated to eliminate the interference from the extraction process. Affected samples must be re-extracted and re-analyzed.

4.1.3 The analyst must take precautions to make sure that contaminants do not enter the analytical system. These precautions include systematic procedures designed to eliminate interferences.

4.2 Some compounds analyzed by this method are unstable or sensitive to extraction and/or instrument conditions:

- Benzidine is easily oxidized during extraction. Neutral extraction may enhance the recovery of this compound.
- Hexachlorocyclopentadiene breaks down photochemically and can decompose from high temperatures, particularly in the injection port of the GC. This compound can also react with acetone in solution.
- 1,2-Diphenylhydrazine is unstable even at room temperature and readily converts to azobenzene.
- Phenols are sensitive to active sites and can give a low response or exhibit poor chromatography by tailing. Therefore, it is important the GC is maintained in the best possible condition. See Section 10.1 for proper daily maintenance.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as 3 and 4-methylphenol.
- Pyridine may perform poorly at the GC injection port temperatures listed in this SOP. Lowering the injection port temperature may reduce the amount of degradation.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Dimethyl-dichloro-silane	Flammable	none	Can be corrosive to the respiratory tract causing severe irritation and tissue damage. Harmful if absorbed through the skin. May cause severe irritation and systemic damage. Severely irritating to the skin and eyes. Harmful if swallowed. Can cause abdominal discomfort, nausea, vomiting, diarrhea, and irritation to the mouth, throat and stomach.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system

6.1.1 Gas chromatograph: An Agilent/HP 6890/7890/900 Intuvo (or equivalent) houses the capillary column. The GC provides a splitless injection port and allows the column to be directly coupled to the mass spectrometer. The oven is temperature programmable to meet the requirements of the method. An HP/Agilent 7673/7683/7963 autosampler (or equivalent) with a 10 ul syringe provides automatic injection of sample extracts while the instrument is unattended.

6.1.2 Analytical Column: 30m x 0.25mm ID, 0.25 um film thickness, Restek Rxi-5Sil MS, Catalog #13623

6.1.3 Mass spectrometer: Agilent (HP) 5972, 5973, 5975 or 5977A Mass Selective Detector (MSD) Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts electron energy in the electron ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 50 ng of decafluorotriphenylphosphine (DFTPP) which meets the criteria in Section 9.2.1 when 2 ul of the 25 ug/ml GC/MS tuning standard is injected through the GC.

6.1.4 GC/MS interface: Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

6.1.5 Data system: The data system is interfaced to the mass spectrometer and accommodates continuous acquisition and storage of GC/MS data throughout the duration of the chromatographic program. The data system consists of a Hewlett-Packard Chemstation equipped with Mustang software used for instrument control and data acquisition. This, in turn, is interfaced to Eurofins's Chrom software for data processing. Data from sample extract analysis can be accessed in real-time, while sample data reports and library searches can be performed on data files from previously run samples. The software is also capable of searching any GC/MS data file for ions of a specific mass whose abundances can be plotted versus time or scan number which allows integration of abundances for any extracted ion between specified times or scan-number limits. Library searches utilize a NIST 02.1 Mass Spectral Library.

6.2 Bottles, glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

6.3 Injection port liners, splitless

6.4 Injection port septa

6.5 Injection port graphite seals

6.6 Pre-silanized glass wool (Supelco 2-0411 or equivalent)

6.7 Syringes, Assorted sizes 10ul - 1000ul; gas-tight

6.8 Bottles, 10 and 5ml amber screw cap with Teflon liner

6.9 Vials, 2ml amber screw cap with Teflon liner

6.10 Wheaton microvials 100ul (or equivalent)

6.11 Volumetric Flasks, Class A with ground glass stoppers (2ml - 100ml)

6.12 Analytical balance, ASP Model SP-180 (or equivalent), capable of accurately weighing to 0.0001 gr.

7.0 Reagents and Standards

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the MSDS prior to the use of any reagent or standard.

The preparation of standards, surrogates and spiking solutions is documented in the TALS Reagent Module. Formulary reports can be generated upon request.

7.1. Reagents:

7.1.1. Methylene Chloride: J.T.Baker Resi-Analyzed, used for Organic Residue Analysis (P/N 9266-V8 or equivalent).

7.1.2. Methanol: J.T.Baker Purge and Trap Grade (P/N 9077-02 or equivalent).

7.1.3. Sylon-CT: Supelco (P/N 33065-U or equivalent). Sylon-CT is a highly reactive silanizing reagent consisting of 95% Toluene and 5% Dimethyldichlorosilane (DMDCS).

7.1.4. Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2. Standards:

7.2.1. **Calibration Standards (Full Scan Analysis)**: Stock analytical standard solutions are purchased mainly from Restek Corporation. Other standards are prepared in the laboratory as needed using neat compounds or prepared solutions purchased from Agilent, SPEX CertiPrep, Chem Service,

Accustandard, Supelco or other suppliers. Standards prep instructions are detailed for the following full scan analyte list options:

- Full Volume Aqueous Prep; and,
- Reduced Volume Aqueous Prep and Soils

Secondary dilutions are either made from purchased stock solutions as listed below or from prepared solutions as listed in the following table:

NOTE: Second sources (from certified separate lots) are used for ICV standards.

Table 3 – Full Scan Stock Standards			
Target Analyte Standard Name	**Conc. (PPM)**	**Vendor**	**Catalog #**
1,2,3,4-TCDD	50	SPEX	SVO-TANJ-12
Agilent Mix (contains compounds listed in Table 4 below)	2000 *	Agilent	Cus 0456
8270 List 1/ Std #1 Megamix	Varied	Restek	571995
8270 List 1/ Std#9	2000	Restek	569730
8270 List 1/ Std#11	2000	Restek	569732
8270 Surrogate Standard	5000*	Restek	567685
8270 Internal Standard	2000	Restek	567684
8270 List 1/ Std#10	2000	Restek	569731
Bisphenol-A	1000	Agilent	Cus-0457

*Agilent Mix, 8270 list1/std#9 and 8270 Surrogate standard are diluted to 100ppm prior to the preparation of the 1.0ppm and 0.5ppm standards.

Table 4	
Agilent Mix Catalog No. Cus-0456	
Analyte	**Concentration (PPM)**
Pentachloronitrobenzene	2000
2 -tert-butyl-4-Methylphenol	2000
2,6-Di-tert-butyl-4-Methylphenol	2000
Coumarin	2000
Phenyl ether	2000
N,N'-Dimethylaniline	2000
N-Methylaniline	2000
Carbamazepine	2000
Benzonitrile	2000
1,3-Dimethylnaphthalene	2000

7.2.1.1. Individual calibration standards for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 5
Full Volume Aqueous Prep and Soils
Working Standards Preparation

Solution Name	120 PPM	80 PPM	50 PPM	20 PPM	10 PPM	5 PPM	2 PPM	1 PPM	0.5 PPM
8270 List 1/ Std #1 Megamix	3000 ul	2000 ul	2500 ul	500ul	250ul	250ul	100ul	50ul	25ul
8270 List 1/ Std #9	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	50ul	500ul*	250ul *
8270 List 1/ Std #10	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	-	-	-
Agilent custom Mix	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	50ul	500ul*	250ul *
1,2,3,4-TCDD	-	-	500ul	-	-	-	-	-	-
8270 Surrogate Standard	600ul	400ul	500ul	100ul	50ul	50ul	20ul	500ul*	250ul *
8270 Internal Standard	500ul	500ul	1000 ul	500ul	500ul	1000 ul	1000 ul	1000 ul	1000 ul
Bisphenol-A	3000 ul	2000 ul	2500 ul	500ul	250ul	250ul	100ul	-	-
8270 List 1/ Std #11	400ul	300ul	500ul	200ul	125ul	125ul	50ul	25ul	-
Final Volume (ml)	**25**	**25**	**50**	**25**	**25**	**50**	**50**	**50**	**50**

Note: The 1.0ppm and 0.5pmm standards (above) are prepared using the 100ug/ml standard for Agilent custom Mix, 8270 List1/std#9 and 8270 Surrogate Standard.

Table 6
Reduced Volume Extraction/LVI
Working Standards Preparation

Solution Name	24 PPM	16 PPM	10 PPM	4 PPM	2 PPM	1 PPM	0.4 PPM	0.2 PPM	0.1 PPM
120 ppm (see Table 5)	2.0mL								
80 ppm (see Table 5)		2.0 mL							
50 ppm (see Table 5)			2.0 mL						
20 ppm (see Table 5)				2.0 mL					

Table 6 Reduced Volume Extraction/LVI Working Standards Preparation									
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**1 PPM**	**0.4 PPM**	**0.2 PPM**	**0.1 PPM**
10 ppm (see Table 5)					2.0 mL				
5.0 ppm (see Table 5)						2.0 mL			
2.0 ppm (see Table 5)							2.0mL		
1.0 ppm (see Table 5)								2.0 mL	
0.5 ppm (see Table 5)									2.0mL
Final Volume (ml)	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**

7.2.1.2. **Initial Calibration Verification (full scan)**: Second source ICVs for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of ICVs for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 7 8270/625 ICV Working Standards Preparation	
Solution Name	**25 PPM**
8270 List 1/ Std #1 Megamix (2nd Lot)	250ul
8270 List 1/ Std #9 (2nd Lot)	125ul
8270 List 1/ Std #10 (2nd Lot)	125ul
Agilent custom Mix (2nd Lot)	125ul
8270 Internal Standard	200ul
8270 List 1/ Std#11	125ul
Bisphenol-A (2nd Lot)	250ul
Final Volume (ml)	**10**

7.2.1.3. **Surrogate Standards (Full Scan Analysis):** A 5000ppm Surrogate Standard is purchased from Restek for use in spiking blanks, samples and associated QC prior to extraction (reference the applicable sample prep SOPs for spiking instructions).

Table 8 Full Scan Surrogate Standards Solution Restek Catalog No. 567685	
Surrogate Standard Compounds	**Concentration (PPM)**
Nitrobenzene-d5	5000
p-Terphenyl-d14	5000
2,4,6-Tribromophenol	5000
Phenol-d5	5000
2-Fluorobiphenyl	5000
2-Fluorophenol	5000

7.2.1.4. **Internal Standards** (**Full Scan Analysis**): The Internal Standards Solution at 2000ppm is purchased from Restek (Catalog # 567684). The Internal Standard solution is stored in 10ml amber screw cap bottles with Teflon liners in the dark at 4°C. The Internal standard solution is used in preparing all analytical standards. Inject 20ul of this solution (2000ppm) per ml of sample extract prior to analysis resulting in a concentration of 40ppm (ug/ml) in the extract.

Table 9 Full Scan Internal Standards Solution Restek Catalog No. 567684	
Internal Standard Compounds	**Concentration (PPM)**
1,4-Dichlorobenzene-d4	2000
Phenanthrene-d10	2000
Naphthalene-d8	2000
Chrysene-d12	2000
Acenaphthene-d10	2000
Perylene-d12	2000

7.2.2. **Calibration Standards (SIM analysis)**: The Edison lab currently analyzes only a select list of compounds by 8270E SIM (see Sections 1.0 and 2.0). Stock analytical SIM standard solutions are purchased mainly from Agilent. Working standards are prepared from these solutions as listed in the tables in Section 7.2.2.1:

Table 10 Stock SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
Pentachlorophenol	1000ppm	AGILENT	PH-180-1
n-Nitrosodimethylamine	100ppm	AGILENT	NS-100-1
Hexachlorobenzene	100ppm*	AGILENT	CH-151-1
PAH Mix	100ppm	AGILENT	PAH-605-1
Bis(2-chloroethyl)ether	100ppm*	AGILENT	BEC-110-1
4,6-Dinitro-2-methylphenol	1000ppm**	AGILENT	PH-150
1,4-Dioxane	1000ppm**	AGILENT	NV-152-1

*Hexachlorobenzene and Bis(2-chloroethyl)ether are diluted to 10ppm prior to SIM Standards prep

** 4,6-Dinitro-2-methylphenol and 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

NOTE: Second sources (from separate lots are used for ICV standards).

7.2.2.1 Individual calibration standards for SIM analysis are prepared in one of two ways depending upon the technique (full volume aqueous prep or reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 11
Full Volume Aqueous Prep – SIM
Working Standards Preparation

	0.025 PPM	0.05 PPM	0.1 PPM	0.5 PPM	1.0 PPM	5.0 PPM
Pentachlorophenol	2.5uL	2.5uL	12.5uL	10uL	20uL	50uL
n-Nitrosodimethylamine	25uL	25uL	125uL	100uL	200uL	500uL
PAH mix	6.25uL	5uL	25uL	50uL	100uL	200uL
Hexachlorobenzene	25uL	25uL	250uL	1000uL	2000uL	500uL*
Bis(2-chloroethyl)ether	25uL	25uL	250uL	1000uL	2000uL	500uL*
4,6-dinitro-2-methylphenol	50ul	50ul	250ul	200ul	400ul	1000ul
1,4-Dioxane	25ul	50ul	250ul	200ul	400ul	1000ul
ISTD	500uL	200uL	500uL	200uL	200uL	200uL
Final Volume (ml)	**25**	**10**	**25**	**10**	**10**	**10**

*For Hexachlorobenzene and Bis(2-chloroethyl)ether the 5.0 ppm level is prepared using the 100ppm standard.

Table 12
Reduced Volume Extraction/LVI – SIM
Working Standards Preparation

	0.005 PPM	0.01 PPM	0.02 PPM	0.10 PPM	0.20 PPM	1.0 PPM
0.025 PPM Std (see Table 11)	1.0 mL					
0.05 PPM Std (see Table 11)		1.0 mL				
0.1 PPM Std (see Table 11)			1.0 mL			
0.5 PPM Std (see Table 11)				1.0 mL		
1.0 PPM Std (see Table 11)					1.0 mL	
5.0 PPM Std (see Table 11)						1.0 mL
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**

7.2.2.2 Initial Calibration Verification (SIM): A 0.1 ppm separate lot SIM ICV is prepared as detailed in Table 13 using the stock standards detailed in Section 7.2.2 (above)

Table 13 0.1ppm SIM ICV preparation	
Pentachlorophenol	25uL
n-Nitrosodimethylamine	25uL
PAH mix	5uL
Hexachlorobenzene	5uL
1,4-Dioxane	5ul
4,6-Dinitro-2-methylphenol	100ul
ISTD	**100uL**
Final Volume	**5 ml**

7.2.2.3 Internal Standard solution (SIM): A 50 ppm Internal Standard solution for SIM analysis is prepared by adding 125ul of the 2000ppm stock ISTD (see Section 7.2.1.4) and bringing to volume with Methylene Chloride in a 5ml volumetric flask.

7.2.2.3.1 For SIM analysis inject 20ul of this solution (50ppm) per ml of sample extract prior to analysis resulting in a concentration of 1ppm (ug/ml) in the extract.

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7.2.3. Calibration Standards (Isotope Dilution SIM – 1,4-Dioxane): The Edison lab currently analyzes only for 1,4-dioxane by 8270E isotope dilution SIM (see Sections 1.0 and 2.0). Stock analytical isotope dilution SIM standard solutions are purchased mainly from Accustandard and Restek. Working standards are prepared from these solutions as listed in the tables below.

Table 14 - Stock 1,4-Dioxane Isotope Dilution SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dioxane	1000ppm*	Accustandard	APP-9-096

* 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

Table 15 - Stock Labeled 1,4-Dioxane SIM Surrogate/Internal Standard (added at prep)			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dioxane-d8	2000ppm	Restek	30614

Table 16 - Stock 1,4-Dioxane Isotope Dilution SIM Internal Standard (added to extract)			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dichlorobenzene-d4	2000ppm	Accustandard	AZ-014J-3

7.2.3.1 Individual calibration standards for 1,4-dioxane isotope dilution SIM analysis are prepared at the concentrations detailed in the following tables. Prepare by combining the appropriate volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 17 Reduced Volume Extraction/LVI – 1,4-Dioxane Isotope Dilution SIM ICAL Standard Concentrations (ug/ml)									
	Lev 1	**Lev 2**	**Lev 3**	**Lev 4**	**Lev 5**	**Lev 6**	**Lev 7**	**Lev 8**	**ICV***
1,4-Dioxane	0.02	0.04	0.1	0.2	0.5	1	2	10	0.2
1,4-Dioxane-d8	4	4	4	4	4	4	4	4	4
1,4-Dichlorobenzene-d4	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2

***:** The ICV is prepared from the second source stock in Table 13.

7.2.4. GC/MS Instrument Performance Check (DFTPP): The DFTPP standard is prepared by is prepared at 25 ppm by adding 2.5ml of EPA 8270 GC/MS Tuning Solution II (Restek Catalog # 31615) to a 100ml volumetric flask and bringing to volume with Methylene Chloride.

7.2.5. Information on prepared standard solutions must be recorded in the TALS Reagent Module. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Standards must be remade every 6 months, or sooner, if the standards expire or begin to show signs of unacceptable degradation. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.6. Please refer to Eurofins Edison SOP No. ED-GEN-008, Standard Operating Procedure for Preparation, Purity and storage of Reagents and Standards.

- Shelf Life of Standard: 1 year after preparation or stock standard manufacture expiration, whichever comes first;
- Storage Requirements: Stock standards are stored at 4°C and Working Standards stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

8.2 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.3 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.4 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 1L	1000 ml or 250 ml (1)	Cool 4 ± 2ºC	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270E
Solids	Wide mouth glass, 8 or 16 oz.	50g	Cool 4 ± 2ºC	14 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270E

(1) : Reduced volume extraction (RVE) LVI option

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every sample	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are determined annually and are updated into TALS limit group..

9.1.1. Method blanks are extracted with every sample batch on each day that samples are extracted. To be considered acceptable, the method blank must contain less than the reporting limit of all target compounds except for phthalates, which can be present at up to 5x the MDL. For method 8270E the method blank is generally acceptable if target analyte concentrations are less than the one half the reporting limit.

If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed.

9.1.1.1. Surrogate recoveries for the method blank are compared to laboratory generated limits. If two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference.. If any surrogate is still outside limits, all samples and QC samples associated with that method blank must be re-extracted (volume permitting).

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared and extracted concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria. See the current active TALS 8270 Method Limit Group for QC limits. The MS/MSD spiking solution should the same as used for the calibration standards.

9.1.2.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.2.2 An LCS/LCSD may be substituted for the MS/MSD if insufficient sample volume is available.

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (See the current active TALS 8270 Method Limit Group for QC limits). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.3.2 Spike recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.4. Surrogate Standards: All full scan samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.1.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (See the current active TALS 8270 Method Limit Group for QC limits).

If any two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.

9.1.4.1 Surrogate recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.5. Internal Standards: The response (area count) of each internal standard in the sample must be within -50 +100% of its corresponding internal standard in the CCV or, the ICAL midpoint for samples analyzed under the initial calibration range. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.2. Instrument QC

9.2.1 GC/MS Instrument Performance Check (DFTPP): (**Note**: the DFTPP performance check applies only to full scan analyses and is not evaluated for SIM analysis). The GC/MS system is tuned using Perfluortributylamine

(PFTBA) such that an injection of 50ng of Decafluorotriphenylphosphine (DFTPP) meet the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all DFTPP key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples. Daily tune verification is not required for 8270E CCV.

DFTPP Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
51	30-60% of mass 198
68	<2% of mass 69
69	reference only
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base Peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

9.2.1.1. Evaluate DFTPP using three scan averaging and background subtraction techniques. Select the scan at the peak apex, add +1 scan from the apex and -1 scans from the apex.

9.2.1.2. The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions. Background subtract DFTPP by selecting a scan for subtraction ≤20 scans before the apex scan of DFTPP.

9.2.1.3. Check column performance using pentachlorophenol and the benzidine peaks (these compounds are included in the DFTPP solution). Benzidine & Pentachlorophenol should respond normally without significant peak tailing (Tailing Factor should be <2 measured at 10% peak height). If responses are poor and excessive peak tailing is present, corrective action for the GC/MS instrument may be required. Corrective actions may include:

9.2.1.3.1 Retune the GC/MS;
9.2.1.3.2 Clip the injector end of the GC column;
9.2.1.3.3 Replace the septum and injection port liner;
9.2.1.3.4 Change the injection port seal;

9.2.1.3.5 Replace the GC column;
9.2.1.3.6 Clean the injection port with MeCl2
9.2.1.3.7 Clean the MS ion source;
9.2.1.3.8 Place a service call.

9.2.1.4. The breakdown of 4, 4-DDT into 4,4-DDD and 4,4'DDE may also be used to assess GC column performance and injection port inertness. If so evaluated the breakdown must be <20%.

9.2.1.5. DFTPP parameter settings are stored in a tune file, which will be used in all subsequent analysis of standards and sample extracts.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. Initial Calibration: The initial calibration range consists of a minimum of five concentration levels of analytical standards (six for second order regression) prepared as described in Section 7.2. and analyzed once the DFTPP instrument performance check has met the criteria in Section 9.2.1. .

9.2.2.2. Initial Calibration Verification (ICV): An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2. The ICV must be from a source (or lot) separate from the standards used in the Initial Calibration Range.

9.2.3 Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV): A mid-point Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the DFTPP instrument performance check (when applicable).. The CCV is prepared as detailed in Section 7.2. (typically, 50 ug/ml for full volume aqueous and soils, 10 ug/ml for LV, 0.02 ug/ml for LVI SIM) and 0.2 for isotope dilution SIM). Additionally a Low Level Continuing Calibration Verification (LLCCV) is analyzed after the CCV for full scan analysis. The LLCCV is the same as the lowest calibration level analyzed with the initial calibration range (See Section 7.2).

9.2.4 Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. Obtain the retention time for all compounds from the analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window by using the absolute retention time for each analyte, internal standard and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the

same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. For qualitative identification to be acceptable the retention time of the relative retention time (automatically calculated in Chrom) must be within 0.8 - 1.2 RRT units of its assigned internal standard. The relative retention times of each compound in the five calibration standards must agree within .06 relative retention time units.

9.2.4.2. Initial Calibration Range: Internal standard calibration is employed for this method. After the initial calibration range has been analyzed the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_x = Area characteristic ion (see Table 21) for the compound
Ais = Area characteristic ion (see Table 21) of associated internal standard
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound. Minimum response factors must be met for each of the compounds listed in Table 18 (below). Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity in the analytical batch to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met.

Table 18:
Minimum Response Factors

Compound	Minimum Response Factor
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl) ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600

Table 18: Minimum Response Factors	
Compound	**Minimum Response Factor**
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalene	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroanailine	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalene	0.010

Table 18: Minimum Response Factors	
Compound	**Minimum Response Factor**
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010
Pentachloronitrobenzene	0.050

9.2.4.2.2. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

9.2.4.2.3. The % RSD of the RRF's must be ≤20% for each target analyte listed in Table 18. The % RSD of each target analytes must be ≤20% in order for the calibration range to be acceptable. Additionally for 8270E, the calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20%. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit or do not meet the minimum correlation coefficient (0.99) or Relative Standard Error (≤20%) for alternate curve fits (see below) then appropriate corrective maintenance action must be performed. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit **AND** do not meet the minimum correlation coefficient (0.99)) or Relative Standard Error (≤20%) then recalibration is necessary.

9.2.4.2.4. If the above listed criteria is met, the system can be assumed to be linear and sample analysis may begin and the average RF from the initial calibration range is used to quantitate all samples.

9.2.4.2.4.1 Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.2.5. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.5.4 Calculate the first order linear regression for any compound which did not meet the 20% criteria. First order linear regression calibration may be employed if alternative average response calibration procedures were not applicable. The r value (Correlation Coefficient) of the equation must be ≥0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.5.2 Second order regression calibration can be used for any compound that has an established history as a non-linear performer.

9.2.4.2.5.3 If second order regression calibration is used a minimum of six (6) calibration levels must be analyzed.

9.2.4.2.5.4 If second order regression calibration is used, the r^2 (Correlation Coefficient) value must be ≥ 0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.5.5 Any compound that fails to meet the 20% RSD or 0.99 correlation coefficient or RSE (≤20%) criteria must be flagged as estimated for detects (or must be noted in the narrative). If there are non-detects the compounds may be reported if there is adequate sensitivity to detect at the quantitation limit. To demonstrate adequate sensitivity analyze the low level point of the initial calibration in each analytical batch (LLCCV) The criteria for demonstrating adequate sensitivity is detection in the LLCCV using the standard qualitative identification criteria.

9.2.4.2.5.6. When calculating the calibration curve using the linear calibration model a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration back into the curve. The recalculated concentration of the low calibration point should be within ±50% of the

standard's concentration. This evaluation can be checked using the Initial Calibration %Drift Report in Chrom. Any detects for analytes calibrated using the linear model and failing this readback criterion must be flagged as estimated or detailed in the narrative.

9.2.4.3. **Calibration Point Read-back Criteria**: Whichever calibration model above is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

- CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD / Linear Response Factor.

- The absolute value of the % Error for each calibration point should be < 30%. For the lowest calibration point, the % Error may be <50%.

- See Section 11.8 for the Calculation of Percent (%) Error.

9.2.4.4. **Initial Calibration Verification (ICV):**.Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 7.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds with the exception of the poor performing compounds listed in Attachment 1 which are allowed to be within 50-150% : An NCM must be initiated to denote any ICV non-conformances.

9.2.4.5. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed these criteria as long as their recoveries are within 65-135%. For the poor performers (see Attachment 1) the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.6. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a DFTPP instrument performance check (not required for 8270E), and analysis of a calibration verification standard. **Note:** Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.5.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of DFTPP. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process. For 8270E analysis only, tune verification is required just prior to ICAL.

9.2.4.5.2 Calibration Verification: Analyze the calibration verification standard immediately after a DFTPP that meets criteria. Daily analysis of the DFTPP is not required as part of the CCV for 8270E analysis. .When samples are analyzed after an ICAL the last ICAL standard may be used as the starting time reference for evaluation. Use the mid point calibration standard (approximately 50ug/l). NOTE: The calibration standard contains internal standards; Dichlorobenzene d_4, Naphthalene d_8, Acenaphthene d_{10}, Phenanthrene d_{10}, Chrysene d_{12}, and Perylene d_{12} at 40ug/l (0.1ug/L for SIM). The calibration check standard must also include all the target analytes from the original calibration.

9.2.4.5.3 The RFs must meet the criteria for the compounds in Table 18. Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met

9.2.4.5.4 The percent difference (when using average response factor) or percent drift (when using linear regression) of the compounds in Table 18 must be ≤20% for at least 80% of the total analyte list. If more than 20% of the compound list fail to 20% difference or drift criterion then appropriate corrective action must be taken prior to the analysis of the samples. Any individual compound that fails must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative identification criteria in the method must be met.

9.2.4.5.5 **CCV Poor Performers**: Refer to Attachment 1 for the identification of poor and/or erratic performing analytes. These analytes are allowed a %D >20% but must be <50 %D to be acceptable. If there are poor performers that exceed 50%D, the data may be reported provided results are noted as estimated. An NCM must be initiated to denote this situation.

9.2.4.5.6 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.5.7 The response (area count) of each internal standard in the calibration verification standard must be within 50 - 100% of its corresponding internal standard in the mid-level calibration standard of the active calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

9.2.4.5.8 The relative retention times of each compound in the calibration verification standard must agree within .06 relative retention time units of its value in the initial calibration.

9.2.4.5.9 Use the average response factors from the original five-point calibration for quantitative analysis of target analytes identified in field samples.

9.2.4.5.10 Prepare a calibration summary or list indicating which compounds did not meet the 20% average percent difference criteria. Record this information in that run log.

9.2.4.7. Low Level Continuing Calibration Verification (LLCCV): An LLCCV consisting of the low level standard from the initial calibration range is analyzed every 12 hours of instrument operation after the CCV. The purpose and evaluation of the LLCCV is described in Section 9.2.4.4.4.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The sequence of events for GC/MS analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed.

10.1.2. Preparation of the Injection Port Liner and Installation Procedure: Prior to the start of initial calibration and each daily analysis of sample extracts, a new liner for the injection port must be prepared. Once a liner has been used it is no longer inert and will cause serious chromatography problems with phenols and other compounds. When preparing the liner, proper laboratory protection must be worn and the liner must be prepared in a well-ventilated hood. When the procedure is completed all traces of toluene, Sylon-Ct and methanol will be removed immediately so that extraction solvents and preparation of sample extracts will not come into contact with these solvents and become contaminated.

10.1.2.1 Remove one liner from a 40ml VOA bottle containing other liners immersed in Sylon-Ct solution. Rinse off the liner with Toluene and wipe dry. Insert 1cm of pre-silanized glass wool partially into one end of the liner and trim neatly. Push the glass wool into the center of the liner so that it is 1 1/4" from the bottom. Do not use glass wool or solvents that are dirty (i.e. suspended particles) or use liners which are chipped on the ends, deformed or fractured. Inspect the glass wool for cleanliness after it has been inserted.

10.1.2.2 Using a Pasteur pipette flush out the interior of the liner containing the glass wool with Sylon-Ct. Rest the liner horizontally on a small beaker and allow the Sylon-Ct to re-deactivate the interior surfaces and the glass wool. There should be no air bubbles caught in the glass wool. After several

minutes flush out the Sylon-Ct with toluene and finally with methanol. Dry the outer surface of the liner and rest it on the injection port housing until the remaining methanol is boiled off

10.1.2.3 Insert the liner with the newly silanized glass wool plug into the injection port. Verify that the column extends up into the injection port and is perpendicular. Inspect the graphite seal and replace it if the edges are knife-shaped.

10.1.2.4 The septum is always replaced daily. Bake out the column at 300°C for 15 minutes after the vacuum in the analyzer has returned to normal.

10.1.2.5 Performance may enhanced by clipping a small portion of the column at the injection port end. Document this activity in the maintenance record.

10.1.3. Prior to calibration or sample analysis always verify that the analyzer is under sufficient vacuum and that the column has proper carrier gas flow.

10.1.4. Establish the following GC/MS operating conditions:

10.1.4.1 Full Scan Operating Mode

Full Scan Mode – Standard Injection Volume
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300°C
Source Temperature: Preset by H.P. at 280°C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45°C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310°C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275°C
Injector: Grob-type, pulse, splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

Full Scan Mode – Large Volume Injection (LVI)
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300°C
Source Temperature: Preset by H.P. at 280°C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45°C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310°C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275°C
Injector: Grob-type, pulse, splitless
Injection Volume: 5ul
Splitless Valve Time: 0.3 minutes

10.1.4.2 SIM Operating Mode

SIM Mode
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300°C
Source Temperature: Preset by H.P. at 280°C
Scan start time: 1.5 minutes
Initial Column Temperature and Hold Time: 40°C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310°C for 3 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275°C
Injector: Grob-type, pulse splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

10.1.4.3 Isotope Dilution Selected Ion Monitoring Mode :

SIM Parameters

Group 1
Plot 1 Ion: 74.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	42.0 50	43. 0 50	68.0 50
	74.0 50	128.0 50	129.0 50
	136.0 50	150.0 50	152.0 50
	93.0 50	66.0 50	
	58.0 50		
	88.0 50		

Group 2
Group Start Time: 6.00
Plot 1 Ion: 152.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	151.0 50	152.0 50	153.0 50
	154.0 50	162.0 50	164.0 50
	165.0 50	166.0 50	

Group 3
Group Start Time: 7.80
Plot 1 Ion: 188.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	94.0 50	101.0 50	142.0 50
	178.0 50	179.0 50	188.0 50
	202.0 50	264.0 50	266.0 50
	284.0 50		

Group 4
Group Start Time: 10.50
Plot 1 Ion: 228

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	120.0 50	228.0 50	229.0 50
	240.0 50		

Group 5
Group Start Time: 12.00
Plot 1 Ion: 252.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	138.0 50	139.0 50	252.0 50
	253.0 50	260.0 50	264.0 50
	267.0 50	276.0 50	278.0 50

Table 19: Target Compound - Primary and Monitoring Ions

Compound	1	2	3

Compound	1	2	3
1,4-Dioxane-d8	96	64	62
1,4-Dioxane	88	58	57
1,4-Dichlorobenzene-d4	152	150	

10.1.5. The above listed instrument conditions are used for all analytical standards for calibration and for all sample extracts analyzed by this method.

10.1.5.1 The column conditions, scan start time, and splitless valve time for analysis of DFTPP only are as follows are as follows:

Initial Column Temperature and Hold Time: 140°C for 0.5 minutes
Column Temperature Program: 140° to 320°C at 22°C/minute
Final Column Temperature Hold: 320C for 0.5 minutes
Scan Start Time: approx. 5 minutes
Splitless Valve Time: 0.3 minutes
Injection Volume: 2 ul

10.2. Analytical Sequence

10.2.1. Dilutions are made based on initial GC/MS analysis. Dilutions are made in 1-ml vials using microsyringes. Calculate the dilution factor using the equation below:

DF= Ph / 5 x Is
Where:
DF = Dilution Factor
Ph = Sample Peak Height
Is = Internal Standard Peak Height

When DF >1 but <2, combine 500ul of sample extract with 500ul methylene chloride in a 1 ml amber vial, add20 ul internal standard and crimp seal

Use **Table 20** to determine dilution and internal standard amount.

Table 20 Dilution Factor Calculations			
DF Value	**Volume of Sample (ul)**	**Volume of Methylene Chloride (ul)**	**Volume of ISTD (ul)**
<1	1,000	None	None
>1, <2	500	500	10
>4, <5	200	800	16
>10, <20	100	900	36
>20	500*	500	10
*Prepare this dilution by serially diluting the >10, <20 dilution			

10.2.2. Instrument Performance and Calibration Sequence

10.2.2.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.2.2.2. Analyze the Instrument Performance Check Standard (DFTPP) as discussed in Section 9.2.1.

10.2.2.3. Initially and as required, analyze the Initial Calibration Range (minimum 5 points, six points for second order regression) as detailed in Sections 7.2.1 and 9.2.4.2. Evaluate the acceptability of the Initial Calibration Range as detailed in Section 9.2.4.2.

10.2.2.4. Immediately after the Initial Calibration Range only, analyze the Initial Calibration Verification (ICV) as detailed in Sections 7.2. and 9.2.4.3. Evaluate the acceptability of the ICV as detailed in Section 9.2.4.3.

10.2.2.5. Every 12 hours, reanalyze and evaluate the Instrument Performance Check Standard (DFTPP), not required for 8270E followed by the Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV) as detailed in Section 9.2.3, 9.2.4.4 and 9.2.4.5. Evaluate the acceptability of the CCV and LLCCV as detailed in Section 9.2.4.4

10.2.2.6. Client samples and QC samples are analyzed (as detailed in Section 10.2.3) after acceptable Instrument Performance and Calibration Checks and until the 12 hour clock expires. Repeat the sequence as required. The automation of GC/MS runs is accomplished via the “SEQUENCE” macro of the ChemStation.

10.2.3. Sample Analysis Sequence

10.2.3.1. Sample extracts are normally prepared on the same day as analysis. The GC/MS operator will prepare the extracts that will be run on his or her instrument. Volume adjustments to the extracts will be made at the discretion of the supervisor.

10.2.3.2. Prior to the start of sample analysis the GC/MS operator will generate a sequence program containing the list of the sample extracts to be analyzed, the position on the autosampler tray, and the proper acquisition and tune methods that are to be used. This sequence program contains all the necessary information on the samples to be analyzed and how the GC/MS system is to analyze them. The sample extracts are loaded onto the autosampler (ALS) tray. Their position is verified by checking them against the ALS number on the sequence. This batch analysis will be performed automatically over the 12-hour period.

10.2.3.3. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.3. Data Processing

10.3.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled. Chrom's auto-processing system queries TALS (LIMS) for each sample's processing parameters (including target compounds lists) and downloads the required processing methods from LIMS to analyze data. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.3.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.3.3. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW 8270E are listed in Table 21.

10.4. Interpretation and Qualitative Identification: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The

characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.4.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.4.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.4.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.4.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.4.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.4.1.6. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

10.4.1.7. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.4.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.4.2.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.4.2.2. The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

10.4.2.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.4.2.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.4.2.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.4.2.6. If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc.).

10.5. Data Reporting

10.5.1. Final Report. The Chom data system automatically produces a data report consisting of hardcopy reports corresponding to specific data reporting requirements, which is uploaded to the TALS LIMS System for the report production group.

10.5.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.5.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.5.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.5.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.5.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.5.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

10.6. The low-level calibration standard establishes the reporting limit. All reported data must be at a concentration at or above the low concentration standard. Any quantitative values below the report limit must be qualified as estimated.

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11.0. Calculations/Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method (see the formula in Section 11.3).

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3. See Section 9.2.4 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method (see formula in Section 11.3). For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Internal Standard Calculation:

11.3.1. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2. Solid Samples

$$\text{Concentration } (\mu g/KG) = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area characteristic ion for the compound (see Table 21)
Ais = Area characteristic ion of associated internal std (See Table 21)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.4 (Initial calibration):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\% D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.8. Calculation of Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level

11.9. Calculation of Percent (%) Error

$$\%Error = \frac{x_i - x_i'}{x_i} x\,100$$

Where:

x_i ' = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount

11.10. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the Eurofins corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Lower Limit of Quantitation Verification

The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-

up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure.

12.3.1 The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all of the same preparation and clean-up methods as client samples.

12.3.2 The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand. Other clean matrices may be used in addition, for project specific requirements.

12.3.3 The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3.4 Recovery of each analyte must meet the laboratory established LCS recovery limits + 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery.

NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0. Pollution Control

13.1 It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

14.1. Pollution Prevention

14.2.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

14.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method SW8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Laboratory Manual, Physical/Chemical Methods, Revision 5, July 2014..

15.2. United States Environmental Protection Agency, “Method SW8270E, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Update IV, Laboratory Manual, Physical/Chemical Methods, Revision 6, June 2018.

15.3. United States Environmental Protection Agency, "Method SW8000D: Determinative Chromatographic Separations", Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Update V, Revision 4, October 2012..

15.4. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.5. Eurofins Edison SOP No. ED-ORP-002, *SW846 Method 3510C-Extraction of Semi-Volatile Organic Compounds in Water by Separatory Funnel,* current revision.

15.6. Eurofins Edison SOP No. ED-ORP-043, *SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270,* current revision.

15.7. Eurofins Edison SOP No. ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW3546, current revision.*

15.8. Eurofins Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.9. Eurofins Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.10. Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.11. Eurofins Edison SOP No. ED-GCS-001, *Preparation and Screening of Semivolatile Organic Extracts for GC/MS Analysis*, current revision.

15.12. Eurofins Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.13. Eurofins Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision.

15.14. Eurofins Edison SOP No. ED-ORP-001, *Extraction of Semivolatile Organic Compounds in Water, EPA Method 625.1,* current revision.

15.15. Eurofins Edison SOP No. ED-GEN-022, *Training,* current revision.

15.16. Eurofins Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

15.17. Eurofins Corporate Quality SOP No. CA-Q-S-006, *Detection and Quantitation Limits,* current revision.

16.0. Method Modifications:

Method 8270E requires the DFTPP tune standard to be analyzed once prior to an ICAL

and not daily prior to sample analysis. Until such time as 8270D is removed from lab capabilities and in order to satisfy both 8270D and 8270E The laboratory will analyze the DFTPP tune daily, prior to QC and sample analysis. The laboratory will use the tighter criteria from Methods 8270C/8270D for tune evaluation, rather than the criteria suggested in Table 3 of Method 8270E.

17.0. Attachments

Attachment 1 Poor Performing Analytes

18.0. Revision History

- Revision 10, date 10/18/2022
    - Updated to Eurofins branding throughout.
    - Section 8.1: updated to include procedure for sampling chlorinated water sources.
    - Section 9 updated throughout to include ICAL evaluation with Relative Standard Error (RSE).
    - Section 11.8: added formula for calculation of Relative Standard Error (RSE)

- Revision 9, date 03/15/2021
    - Updated as needed to reflect 1,4-dioxane RL of 0.2 ug/l.
    - Updated Tables 11 and 12 to reflect new low ICAL standard concentration of 1,4-dioxane..

- Revision 8, date 06/29/2020
    - Updated to Eurofins branding.
    - Updated throughout to include 8270E requirements.
    - Removed references to SW846 3550B/C prep methods (no longer in use for this method at Edison lab).
    - Update equipment listed in Section 6.0.Updated analytical column in Section 6.1.2.
    - Updated, deleted and renumbered tables as required.
    - Made extensive updates to Standards (sources and preparation) in Section 7.2.
    - Removed all references to Aromatic Amines. Deleted all tables specific to Aromatic Amine analysis. Renumbered remaining tables in document and updated text references.
    - Throughout document clarified tune requirements for 8270E.
    - Following added to Section 9.1.1: For method 8270E the method blank is generally acceptable if target analyte concentrations are less than the one half the reporting limit.
    - Calibration Point Read-back Criteria was added to Section 9.2.4.3. The calculation for percent error was added to Section 11.8.
    - Section 9.2.4.2.3: added following for 8270E: the calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20%.
    - Section 9.2.4.2.5.6: added ‘The recalculated concentration of the low calibration point should be within ±50% of the standard’s concentration.”

- Section 12.1 revised to reflect the updated MDL procedure.
- Added Section 12.3: annual Lower Limit of Quantitation Verification
- Added Corporate SOP CA-Q-S-006, Detection and Quantitation Limits to references.
- Section 16.0: added a Method Modification regarding tuning check requirements.

- Revision 7, date 06/08/2018

  - Section 2.3: revised to clarify that RVE/LVI is lab standard procedure.
  - Section 9.1.3: removed statement regarding allowance for up to five analytes to recover outside of lab acceptance limits in LCS/LCSD.
  - Section 9.2.4.3: Replace table 'ICV Poor Performers (50-150% Recovery) with expanded list of 'Poor Performing Analytes' in Attachment 1.
  - Added Section 9.2.4.4.5: CCV Poor Performers
  - Corrected number in section 9.2.4.5
  - Added Attachment 1 – Poor Performing Analytes

- Revision 6, date 01/12/2018:

  - Section 7.2.5 included to specify reagent and standard storage conditions.
  - Revised Section 9.1.3 to clarify requirements for specific LCS/LCSD evaluation criteria regarding the # of out of criteria analytes.
  - Revised Section 9.2.4.3 to add 2,4-Dimethylphenol as a poor performing analyte, increased the range for the poor performers to 50-150 and also expanded the guidelines for flagging the ICV outliers.

- Revision 5, dated 09/29/2017:
  - Revised Section 9.1.1 to clarify requirements for surrogate recovery in method blanks.

- Revision 4, dated 08/21/2017:
  - Updated throughout to add a procedure for the analysis of 1,4-dioxane by isotope dilution selected ion monitoring (SIM)
  - Added tables for isotope dilution SIM standards. Renumbered all tables as necessary.
  - Section 7.2.1: added a list of full scan calibration list options.
  - Table 3: Renamed 'Full Scan Stock Standards'.
  - Section 9.2.1: noted that DFTTP applies only to full scan analysis.
  - Section 9.2.3: updated CCV concentrations
  - Added reference to GC/MS Tuning Policy in Section 15.16.

- Revision 3, dated 01/07/2016:
  - Tables 1 and 2: added SIM as option for 1,4-Dioxane.
  - Section 2.3: removed SW3541 (Soxtherm) as option for soils prep (lab has discontinued use of this method). Also removed SW3541 SOP reference from Section 15.0.
  - Tables 19 and 20: added source and prep instructions for 1,4-Dioxane SIM standard. Updated source and prep instructions for 4,6-Dinitro-2-methylphenol.
  - Table 22: added prep instructions for 1,4-Dioxane and 4,6-Dinitro-2-methylphenol

SIM ICV standard.
- Corrected the information in the 'DFTPP Key Ions and Abundance Criteria' table in Section 9.2.1 to match the info found in SW846 8270C.
- Section 10.1.4.2: updated "SIM Parameters" to included ion masses/dwell times for 1,4-Dioxane.

- Revision 2, dated 01/28/2015:
  - Extensively reformatted the SOP. Placed tables that had been in rear of document into the body of the text. Renumbered tables as applicable and fixed text references to tables.
  - Section 1.1, Table 1: Revised table to include all current analytes. Also footnoted those compounds which are currently analyzed by SIM.
  - Section 2.3: added options for extraction of solids by SW846 3456 (Microwave Extraction) and by SW3580A (Waste Dilution) and added SOP references. Deleted reference to SOP ED-ORP-005 (SW3550B – Low Level); Updated Section 15 (References).
  - Section 2.5: added text detailing the RVE/LVI options.
  - Section 2.6: added table which includes all analytes routinely analyzed by SIM.
  - Section 6: updated to include newer GC, MS and autosampler models currently in use.
  - Section 6.1.3: added Zebron ZB column as an option.
  - Section 7.2: extensively revised standards information to reflect switch to Restek standards.
  - Table 3:Added Custom Aromatic Amine Surrogate Standard and revised Table 8 to include initial calibration prep instructions for the Aromatic Amine surrogates.
  - Throughout document: removed references to Target and replaced with Chrom.
  - Section 7.2.1: Added reference to section 10.2.1.2 for LVI.
  - Added Section 7.2.1.3.1 and Table 17A both of which discuss use of Aromatic Amine surrogates.
  - Section 7.2.1.2: Added reference to Tables 9,10 and 11 (ICV Preparation)
  - Section 8.0: Added Sample container and minimum sample size (250 ml) for Reduced volume extraction.
  - Sections 9.1.2, 9.1.3, 9.1.4 and 9.2.4: added statement that certain state regulatory programs have defined recovery limits which, where applicable, are used for spike and calibration evaluations.
  - Section 9.1.2: Deleted sentence "A minimum of 16 spiked analytes are reported to in client reports (the full list is reported at least once during each 2 year period because we employ full spiking list.
  - Section 9.1.4: Added note regarding use of Aromatic Amine Surrogates.
  - Section 9.2.2.2: Added reference to ICV Preparation tables in Section 7.2.
  - Section 9.2.3: added more specific info as to the concentration of the CCVs for all techniques.
  - Section 9.2.4.2.1: Changed to reflect that each analyte should meet minimum RF's, not the average across the calibration. Added LLCCV requirement.
  - Section 10.3.1: added explanation of Chrom's interaction with TALS. Removed references to Target.
  - Section 9.2.4.2.5.5: Added: (or can be noted in the narrative)
  - Section 9.2.4.2.5.6: Revised last sentence to read: "This evaluation can be checked using the Initial Calibration %Drift Report in Chrom."
  - Section 9.2.4.3: Removed 65-135% criteria and added "poor performing" analyte list and associated criteria of 60-140%.

- Section 9.2.4.4.3: Added LLCCV criterion for RFs
- Section 9.2.4.4.4: Added LLCCV criterion for %D
- Section 10.1.4: Updated GC/MS operating conditions for full scan, SIM and DFTPP.
- Section 10.1.4.1: added a table detailing operating conditions for LVI option.
- Table 2: Added 2-ethylaniline, 2,4-dimethylaniline, 3,4-dimethylaniline, 2,3-dimethylaniline, 2,4,5-trimethylaniline and 4-chloro-o-toluidine to Working Standards preparation information.
- Table 25: updated to include all current analytis/surrogates/internal standards and associated ions.
- Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision.

- Revision 1, dated 11/07/2011

  - Section 1.1, Table 1: Added Pentachloronitrobenzene and associated CAS# to the analyte list.
  - Section 7.2.1: Added Pentachloronitrobenzene standard information.
  - Table 2: Added Pentachloronitrobenzene to Working Standards preparation information.
  - Table 4: Added Pentachloronitrobenzene and associated minimum RF.
  - Table 8: Added Pentachloronitrobenzene and associated ions.

- Revision 0, dated 02/22/2011: NEW

Table 21
Characteristic Ions Of Semi-Volatile Organic Compounds

Compound	Primary Ion	Secondary Ion(s)
1,1'-Biphenyl	154	153,76
1,2,4,5-Tetrachlorobenzene	216	214, 179
1,2,4-Trichlorobenzene	180	182, 145
1,2-Dichlorobenzene	146	148, 111
1,2-Diphenylhydrazine	77	105, 182
1,3-Dichlorobenzene	146	148, 111
1,3-Dimethylnaphthalene	156	141, 115
1,4-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene d4 **(ISTD)**	152	150, 115
1,4-Dioxane	88	58, 43
1-Methylnaphthalene	142	141, 115
1-Naphthylamine	143	115, 116
2,2'-oxybis[1-chloropropane]	45	77, 121
2,3,4,6-Tetrachlorophenol	232	131, 230
2,3,7,8-TCDD (screen)	320	322, 324
2,3-Dihydroindene		
2,3-Dimethylaniline	106	129

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
2,4,5-Trichlorophenol	196	198, 200
2,4,5-Trimethylaniline	102	55, 56
2,4,6-Tribromophenol **(Surrogate)**	330	132, 141
2,4,6-Trichlorophenol	196	198, 200
2,4-Dichlorophenol	162	164, 98
2,4-Xylidine	121	120, 106
2,4-Dimethylphenol	122	107, 121
2,4-Dinitrophenol	184	63, 154
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
2-Chloronaphthalene	162	127, 164
2-Chlorophenol	128	64, 130
2-Ethylaniline	106	122,104
2-Fluorobiphenyl **(Surrogate)**	172	171
2-Fluorophenol **(Surrogate)**	112	64
2-Methylnaphthalene	142	141
2-Methylphenol	108	107
2-Naphthylamine	143	115, 116
2-Nitroaniline	65	108, 138
2-Nitrophenol	139	109, 65
2-tert-butyl-4-Methylphenol	149	121, 91
2-Toluidine	107	106, 77
3,3'-Dichlorobenzidine	252	254, 126
3,4-Dimethylaniline	106	129, 127
3,5-Di-tert-butyl-4-Hydroxytol	205	220, 145
3-Nitroaniline	138	108, 65
4,6-Dinitro-2-methylphenol	198	51, 105
4-Bromophenyl phenyl ether	248	250, 141
4-chloro-2-methylaniline	106	144, 142
4-Chloro-3-methylphenol	107	144, 142
4-Chloroaniline	127	129
4-Chloroaniline-d4 **(Surrogate)**	131	133
4-Chlorophenyl phenyl ether	204	206, 141
4-Methylphenol	108	107
4-Nitroaniline	138	108, 65
4-Nitrophenol	139	109, 65
Acenaphthene	154	153, 152
Acenaphthene d10 **(ISTD)**	164	162, 160
Acenaphthylene	152	151, 153
Acetophenone	105	77, 51
Aniline	93	66
Aniline-d5 (**Surrogate**)	98	71,42
Anthracene	178	176, 179
Atrazine	200	173,215
Benzaldehyde	77	105,106
Benzidine	184	92, 185

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic Acid	122	105, 77
Benzyl Alcohol	108	79, 77
Bis(2-chloroethoxy)methane	93	95, 123
Bis(2-chloroethyl)ether	93	63, 95
Bis(2-ethylhexyl)phthalate	149	167, 279
Bisphenol-A	213	228, 119
Butyl benzyl phthalate	149	91, 206
Caprolactam	113	55,56
Carbamazepine	193	236, 135
Carbazole	167	166, 139
Chrysene	228	226, 229
Chrysene d12 **(ISTD)**	240	120, 136
Coumarin	146	118, 63
Dibenz(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
Diethylphthalate	149	177, 150
Dimethylphthalate	163	194, 164
Di-n-butylphthalate	149	150, 104
Di-n-octylphthalate	149	167, 43
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Hexachlorobenzene	284	142, 249
Hexachlorobutadiene	225	223, 227
Hexachlorocyclopentadiene	237	235, 272
Hexachloroethane	117	201, 199
Indeno(1,2,3-cd)pyrene	276	138, 227
Isophorone	82	95,138
Kepone	272	237, 355
N,N-Dimethylaniline	120	122, 104
Naphthalene	128	129, 127
Naphthalene d8 **(ISTD)**	136	68
n-decane	43	57
Nitrobenzene	77	123, 65
Nitrobenzene-d5 **(Surrogate)**	82	128, 54
N-Nitrosodimethylamine	42	74, 44
N-Nitroso-di-n-propylamine	170	42,101,130
N-Nitrosodiphenylamine	169	168, 167
n-Octadecane	57	43, 85
o-Toluidine-d9 **(Surrogate)**	114	112, 42
Pentachloronitrobenzene	237	214,295
Pentachlorophenol	266	264, 268

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
Perylene d12 **(ISTD)**	264	260, 265
Phenanthrene	178	179, 176
Phenanthrene d10 **(ISTD)**	188	94, 80
Phenol	94	65, 66
Phenol-d5 **(Surrogate)**	99	42, 71
Phenyl ether	170	77, 115
Pyrene	202	200, 203
Pyridine	79	52, 51
Terphenyl-d14 **(Surrogate)**	244	122, 212

Attachment 1
Poor Performing Compounds

1,2,4,5-Tetrachlorobenzene
1,4-Dioxane
1-Naphthylamine
2,3,4,6-Tetrachlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
2-Chloroaniline
2-Naphthylamine
3&4-Methylphenol
3'3-Dichlorobenzidine
4,6-Dinitro-2-methyl- phenol
4-Chloroaniline
4-Nitrophenol
Aniline
Atrazine
Benzaldehyde
Benzidine
Benzoic Acid
Benzyl Alcohol
Biphenyl
Caprolactam
Diphenylamine
Hexachlorocyclopentadiene
Hexachloroethane
n-Decane
n-Nitrosodimethylamine
o,o,o-Triethylphosphorothioate
o-Toluidine
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

These analytes are exempt from the ICV and CCV criteria as detailed in this SOP

Title: Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A

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Approvals (Signature/Date):

06/05/2024		06/05/2024	
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06/05/2024		06/05/2024	
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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits: SW846 Method 8151A provides extraction and derivatization procedures for the gas chromatographic analysis of chlorinated acid herbicides in water, soil, and waste samples. An option for the hydrolysis of esters is also described. This SOP details the extraction of soil samples with diethyl ether follower by esterification with diazomethane (or diazald) The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to Eurofins Edison SOP No. ED-GCS-005, Analysis of Organochlorine Herbicides by SW846 Method 8151A, current revision.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 9 (Service to the Client) and 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

This SOP provides extraction and derivatization procedures for the determination of chlorinated acid herbicides and their esters in solid samples. Soil samples are serially extracted with diethyl ether. The extracts are hydrolyzed, dried and concentrated and then esterified with Trimethylsilyl diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The sensitivity of Method 8151 depends on the level of interferences in addition to instrumental limitations.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing reagent blanks.

Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by rinsing it with the last solvent used in it. This should be followed by detergent washing with hot water and rinses with tap water, then with organic-free reagent water. Glassware should be solvent-rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used.

Matrix interference may be caused by contaminants that are co-extracted from the sample. Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

The acid forms of the analytes are strong organic acids that react readily with alkaline substances and can be lost during sample preparation. Glassware and glass wool must be acid-rinsed with 1 N hydrochloric acid and the sodium sulfate must be acidified with sulfuric acid prior to use to avoid analyte losses due to adsorption.

Sample extracts must be dry prior to methylation or else poor recoveries will result.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Ethyl Ether is an extremely flammable solvent. If a mechanical device is used for sample extraction, the device should be equipped with an explosion proof motor and placed in a hood.

Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.

Diazomethane can explode under certain conditions therefore take the following precautions:

- Use a well-ventilated hood.
- Use a safety screen.
- Do not heat above 90 degree Celsius.
- Store away from alkali metals.
- Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

Ether creates excessive pressure very rapidly. Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of within six months.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Equipment

- Top loading Balance - Sargent Welch S2642-25 or equivalent
- N-Evap - Meyer Analytical Evaporator Model No. 112 or equivalent
- Explosion Proof Hood

6.2. Supplies

- 250 ml Clear Glass Jar
- 500 ml Erlenmeyer Flask
- 125 ml Erlenmeyer Flask
- 125 ml Separatory Funnel
- 10 ml graduated Concentrator Tube - Kontes K570050-1025 or equivalent.
- 10 ml glass vials with Teflon lined screw caps.

- 20 ml glass vials with Teflon lined screw caps.
- 5 ml to 1000 ml Class A Volumetric flasks
- Funnel - 75 mm diameter
- 50 ul Gastight Syringe - VWR 60376-241 or equivalent.
- 100 ul Gastight Syringe - Baxter S9662-5 or equivalent.
- 100 ml Graduated Cylinder
- 1 ml Pasteur Disposable Pipette
- 10 ml Disposable Pipette
- Acid Washed Glass Wool – Fisher cat # B0773KMDPK or equivalent.
- pH Paper
- Tongue Depressors
- EM Quant Peroxide Test Strips, EMD No. 10011-1 or equivalent

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in Eurofins Quality SOP No. CA-Q-S-001 (Solvent & Acid Lot Testing & Approval) and Eurofins Edison SOP No. ED-GEN-023 (Bulk Solvent Testing and Approval).

- Trimethylsilyl diazomethane (TMSD), 2.0 molar solution in hexane, Acros 429211000 or equivalent.
- Diethyl Ether (Residue Analysis Grade with 2% Ethanol preservative) - Baker 9259-03 or equivalent (tested for peroxide formation upon opening and monthly thereafter by use of EM Quant Peroxide Test Strips)
- Methylene Chloride (High Purity Grade) - Baker 9266 or equivalent.
- Acetone (High Purity Grade) – Baker 9254 or equivalent.

- Sodium Sulfate Anhydrous Crystals (Reagent Grade) – Fisher or equivalent. (Baked at 400□C for 4 hours)
- Hexane (High Purity Grade) – Baker 9262 or equivalent.
- Potassium Hydroxide (Analytical Reagent Grade) – Aldrich 306568 or equivalent
- Sulfuric Acid (Analytical Reagent Grade) – FisherA300S212 or equivalent.
- Peroxide Test Strips - Aldrich Z10, 168-0 or equivalent
- Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At Eurofins Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).
- Diazald - Aldrich D2, 800-0 or equivalent.

7.1.1 Reagent preparation

7.1.1.1 Anhydrous sodium sulfate crystals must be baked in the muffle furnace at 400 degrees C for 4 hours prior to use. After baking, the sodium sulfate must be acidified. Place 500 grams of anhydrous sodium sulfate into a 500 ml wide mouth Erlenmeyer flask. Add enough diethyl ether to cover the sodium sulfate. Add 0.5 ml concentrated sulfuric acid. Shake vigorously for 1 minute. Evaporate the ether by placing the flask on steam bath. Mix 1 g of the resulting solid with 5 ml of organic-free reagent water and measure the pH of the mixture. It must be below a pH of 4. Prepare fresh daily.

7.1.1.2 37% KOH Solution: Rinse a 1 liter Pyrex flask three times with organic free reagent water. Weigh 148 g KOH into flask and add 400 ml organic free reagent water. Mix until the KOH is completely dissolved and the flask has cooled to room temperature. Transfer the KOH solution to a clean 500-ml amber bottle and label.

7.1.1.3 12N H2SO4: Rinse a 1 liter Pyrex flask three times with organic free reagent water. Add 200 ml organic free

reagent water to the flask. Slowly, and while mixing, add 100-ml concentrated H2SO4 to flask. Continue mixing until flask has cooled to room temperature. Transfer the H2SO4 solution to a clean 500-ml amber bottle and label.

7.1.1.4 25% H2SO4 solution: 25 ml Acid and 75 ml reagent water. Add 75 ml organic free reagent water to a graduated cylinder. Slowly, and while mixing, add 25-ml concentrated H2SO4 to cylinder. Continue mixing until cylinder has cooled to room temperature. Transfer the H2SO4 solution to a clean amber bottle and label.

7.2 Standards

7.2.1 Standards are purchased as concentrated solutions (see Section 7.2.2). Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.3.

7.2.2 Standard mixes and sources: Table 1 lists the standard concentrate mix sources. Table 2 details the individual components of these mixes.

Table 1:
Herbicide Standard Mixes and Sources*

Standard Name ("Lab Name")	Concentration (ug/ml)	Source
Acid Herbicide LCS RTS ("Spiking Mix")	varies (in Methanol)	Restek Catalog # 567950
DCAA standard	1000 (in methanol)	Restek Catalog #567804

*May be substituted with equivalent standards from alternate sources.

Table 2:
Components of Herbicide Standard Mixes

Parameter	Catalog Nos.	Lab Standard Name	Concentration (ug/ml)
2,4-dichlorophenoxyacetic acid (2,4-D)	Restek Catalog # 567950	Spiking Mix	20
2-(2,4,5-Trichlorophenoxy)propionic acid (2,4,5-TP aka Silvex)	Restek Catalog # 567950	Spiking Mix	5
2,4,5-T	Restek Catalog # 567950	Spiking Mix	5
2,4-DB	Restek Catalog # 567950	Spiking Mix	20
Dalapon	Restek Catalog # 567950	Spiking Mix	20

Table 2: Components of Herbicide Standard Mixes			
Parameter	Catalog Nos.	Lab Standard Name	Concentration (ug/ml)
Dicamba	Restek Catalog # 567950	Spiking Mix	10
Dichloroprop	Restek Catalog # 567950	Spiking Mix	20
Dinoseb	Restek Catalog # 567950	Spiking Mix – 10 compound	20
MCPA acid	Restek Catalog # 567950	Spiking Mix – 10 compound	2000
MCPP acid	Restek Catalog # 567950	Spiking Mix – 10 compound	2000
Pentachlorophenol	Restek Catalog # 567950	Pentachlorophenol spike	5
Picloram (4-amino-3,5,6trichloropicolinic acid)	Restek Catalog # 567950	Picloram Acid Spiking Standard	20
2,4-Dichlorophenylacetic Acid	Restek Catalog # 567804	Surrogate Spiking Solution	1000

7. 3. Standards Preparation

7.3.1. All standard stock solutions are diluted to the working concentrations as required with acetone (as indicated) using Class A volumetric glassware.

7.3.2. Herbicide Spiking Standard (LCS/MS/MSD):

7.3.3.1 Herbicide QC Spiking Standard Mix (12 compound analysis): The (12) compound spiking standard is ready to spike and used when analyzing for the standard list of herbicides The spiking standard mix is a 25 ml solution (Restek Catalog # 567950; see Tables 1 and 2) of the 12 compounds at the specified concentrations as free acids (see Table 2). The concentrate is used without further dilution prior to the spiking of the following quality control sample types: MS, MSD, and LCS. Spiking instructions are found in Section 10.

7.3.3. Herbicide Surrogate Spiking Standard (2,4-DCAA Acid Spike Mix): The herbicide surrogate spiking solution is a 1 ml solution of 2,4-DCAA at a concentration of 1000 ug/ml. (Restek Catalog # 867804,

see Tables 1 and 2). The concentrate is used after 10X dilution in acetone prior to the spiking of samples (final concentration of spiking standard is 100 ug/ml).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soil	Glass	8oz clear glass	Cool 4 ± 2°C	14 days to extraction; 40 days to analysis	USEPA SW846

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2°C) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS) [1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS) [2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD) [2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank

must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess the performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria. If the spiked sample recovery results (MS/MSD) fall outside the laboratory generated limits (refer to the current active TALS method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in Eurofins Edison SOP No. ED-GCS-005, Analysis of Organochlorine Herbicides by SW846 Method 8151A, current revision.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing 2,4-DCAA (see Section 7.3.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

10.0. Procedure

10.1. Sample Extraction and hydrolysis (Soil Samples)

10.1.1. Rinse enough graduated cylinders and flasks once with acetone, twice with diethyl ether and once with 25% sulfuric acid: reagent water.

10.1.2. Weigh out 30g (+/- 0.1g) of soil sample into a clean clear glass 8oz sample container. Mark the lab sample identification numbers on the corresponding sample containers with red wax pencil.

10.1.3. Make up labels for each sample with the following information and place on the corresponding 500 ml Erlenmeyer flasks.

10.1.4. Weigh out approximately 30 g of sodium sulfate into two clean glass jars. These will serve as the blank and LCS samples respectively.

10.1.5. Rinse each syringe to be used for adding surrogates and spikes with acetone.

10.1.6. Add 50 ul of 100 ppm 2,4-Dichlorophenylacetic Acid (DCAA) Surrogate Spiking Solution (see Table 1) to each sample and QC sample. NOTE: Hold the syringe just above the level of the soil when adding the spike. Do not touch the tip of the syringe to the soil or to the side of the glass jar. Swirl the separatory funnel after adding surrogate spike. All spikes must be witnessed.

10.1.7. When preparing QC spike samples (MS/MSD/LCS/LCSD), spike with 250ul of the Herbicide QC Spiking Standard Mix (Restek 569750, see Section 7.3.3.1). Swirl the glass jar after adding the spike. All spikes must be witnessed.

10.1.8. Adjust the pH of each sample and QC sample to 2 with concentrated HCl (about 5ml) and monitor the pH for 15 minutes with occasional stirring. If necessary, add additional HCl until the pH remains at 2.

10.1.9. Add 20 ml of acetone to each sample. Mix each sample with the wrist shaker for 20 minutes.

10.1.10. Add 60 ml of diethyl ether to the each sample and shake again for 20 minutes. Decant the extract and measure the volume of solvent recovered.

10.1.11. Extract the sample twice more using 20 ml of acetone followed by 60 ml of diethyl ether. After addition of each solvent, the mixture should be shaken with the wrist shaker for 10 minutes and the acetone-ether extract decanted.

10.1.12. After the third extraction, the volume of extract recovered should be at least 75% of the total volume of solvent added. If solvent recovery is less than 75%, extract once more with 20 ml of diethyl ether.

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10.1.13. Check the pH of the extract. If it is not at or below pH 2, add more concentrated HCl until stabilized at pH of 2.

10.1.14. Into a pre-rinsed 500ml separatory funnel add 250ml of organic free reagent water.

10.1.15. Add 5ml of H2SO4 to the reagent water and check with pH paper to insure pH is <2.

10.1.16. Add the solvent extract to separatory funnel and gently mix the contents of the separatory funnel for 1 minute and allow the layers to separate.

10.2. Partitioning

10.2.1. Add 2.0 ml of the 37% KOH solution, 15 ml of reagent water.

10.2.1. Allow it to cool for 5-10 minutes.

10.2.2. Pour the extract into a 500-ml separatory funnel pre-rinsed with diethyl ether.

10.2.3. Add 40 ml of diethyl ether to the separatory funnel and shake for two minutes.

10.2.4. Pour the dried diethyl ether extract through a funnel plugged with acid washed glass wool, collecting the extract in the K-D concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.2.5. Repeat twice using 20-ml aliquots of diethyl ether.

10.2.6. Discard the ether phase.

10.2.7. Adjust the pH of the extract remaining in the 500-ml separatory funnel by adding 5 ml of 12N Sulfuric Acid. Check pH with pH paper.

10.2.8. Add 40 ml of diethyl ether to the separatory funnel and shake for 2 minutes with periodic venting to release pressure.

10.2.9. Collect each diethyl ether phase into one pre-cleaned screw top glass jars containing approximately 10 g of acidified sodium sulfate crystals and mix well.

10.2.10. Repeat sections10.2.8 and 10.2.9 twice using 20-ml aliquots of diethyl ether.

10.2.11. Allow the diethyl ether extract to remain in contact with the sodium sulfate for approximately 2 hours.

10.2.12. Pour the dried diethyl ether extract through a funnel plugged with acid washed glass wool, collecting the extract in the Evaporator concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.2.13. Place the white identification tag on the Evaporator apparatus.

10.2.14. Rinse the Erlenmeyer flask and funnel with 20 to 30 ml of diethyl ether to complete the quantitative transfer.

10.4. Extract Concentration

10.4.1. Blow down the sample in the concentrator until the sample volume is approximately 1 ml.

10.4.2. Add 30 ml of hexane and concentrate further on the evaporator to a final volume of approximately 10 ml.

10.4.3. Remove the samples from the evaporator and allow it to drain and cool for at least 10 minutes.

10.4.4 Extracts will be esterified either by TMSD or by Diazald

10.5. Esterification by TMSD

10.5.1. Add approximately 200 uL of methanol to the extract followed by approximately 200 uL of 2.0M Trimethylsilyl diazomethane (TMSD) solution. The extract will turn a yellow color. If this does not occur, add additional (approximately) 200 uL aliquots of 2.0M Trimethylsilyl diazomethane (TMSD) solution until the yellow color persists. Check the sample every 15 minutes for yellow color. If the yellow disappears in this time frame, continue adding an additional aliquot of 2.0M Trimethylsilyldiazomethane (TMSD) solution until the yellow color persists.

10.5.2. Allow the extract to stand for at least one hour at room temperature to allow the methylation reaction to occur. The reaction is halted with the addition of approximately 0.05g (one scoop using a disposable spatula) of silica gel. The extract is then brought up to a final volume of 10 mL with hexane by visually comparing it to a calibrated collection tube.

10.5.3. Adjust the final extract volumes to 10ml with hexane. The extracts ready to be analyzed by GC. Transfer some volume to GC autosampler vials.

10.5.4. Samples must be analyzed as soon as possible (less than 24 hrs. preferably) after esterification. This is to minimize the transesterification and other potential reactions that may occur.

10.5.5. Store refrigerated if further processing will not be performed immediately.

10.5.6. Extracts should be stored at 4 deg C away from light.

10.6 Esterification by Diazald

10.6.1 Add 10 ml of DI water and 15 ml of Carbitol (diethylene glycol monoethyel ether) to a 250-ml Erlenmeyer flask. Dissolve in 6 g of KOH

10.6.2 Prepare a 2-hole rubber stopper holding a 60 ml separatory funnel in one hole, and a length of U-shaped tubing in the other hole. Attach the 2-hole rubber stopper to the Erlenmeyer flask.

10.6.3 Prepare a diazald solution by adding 10 ml of ether to the 60 ml separatory funnel and dissolving in 2 g of diazald.

10.6.4 Place the Erlenmeyer flask/separatory funnel apparatus on a stirring hot plate. Allow the diazald to slowly drip into the Erlenmeyer. The hot plate setting should never exceed 2.

10.6.5 Rinse the end of the U-shaped tubing 3 times with hexane, then place into extract allowing the diazomethane gas to bubble through the extract. The extract should turn a yellow color signaling that esterification is complete.

10.6.6 After esterification, remove excess diazomethane gas by adding a few grams of silicic acid to the extract and bubbling nitrogen gas through the extract on the N-Evap for several minutes'

10.6.7 Adjust the final extract volume of 10 ml with Hexane (5 ml for TCLP). The extracts are ready to be analyzed by GC.

10.6.8 Samples must be analyzed as soon as possible (less than 24 hrs. preferably) after esterification. This is to minimize the trans-esterification and other potential reactions that may occur.

10.6.9 Store refrigerated if further processing will not be performed immediately.

10.6.10 Extracts should be stored at 4 deg C away from light.

10.7. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.7.1. The TALS reagent database must be completed in full with the required information whenever standards are logged and/or prepared.

10.7.2. All required data types (sample volumes, reagent and standard volumes, reagent and standard lots, etc…) must be entered into the TALS batch database each time an extraction is performed.

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11.0. Calculations / Data Reduction

None

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The MDL is the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration or amount is distinguishable from method blank results. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM) consistent with 40CFR Part 136 Appendix B, August, 2017. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to the current revision of Eurofins Edison SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental

management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-182403), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method 8151A: Organochlorine Herbicides by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. Eurofins Edison SOP No. ED- ORP-015, Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A, current revision.

15.3. Eurofins Edison Document No. ED-QA-LQM, Laboratory Quality Manual, current revision.

15.4. Eurofins Corporate Document No. CW-E-M-001, Environmental Health and Safety Manual, current revision.

15.5. Eurofins Corporate Quality SOP No. CA-Q-S-001, Solvent & Acid Lot Testing & Approval, current revision.

15.6. Eurofins Edison SOP No. ED-GEN-023, Bulk Solvent Testing and Approval, current revision.

15.7. Eurofins Edison SOP No. ED-GEN-013, Glassware Cleaning, most current revision

15.8. Eurofins Edison SOP No. ED-GEN-022, Training, current revision.

16.0. Method Modifications:

NONE

17.0. Attachments

NONE

18.0. Revision History

- Revision 13, dated 05 Jun 2024:
  - Removed all KD apparatus no longer used
  - Added the Diazald esterification procedure in case we run out of TMSD.
  - Removed all equipment, reagents, supplies and glassware no longer used.
  - Updated throughout to reflect Eurofins branding.

- Revision 12, dated Oct 08 2020:
  - Section 7.1: added Trimethylsilyldiazomethane (TMSD) , 2.0 molar solution in hexane, Acros 429211000 or equivalent. Removed Diazald..
  - Section 10.5.1: replaced the Diazald esterification procedure with the TMSD procedure.

- Revision 11, dated April 26, 2019
  - Section 10.4.11: methanol was added to extract to help with better herbicide recoveries.

- Revision 10, dated February 22, 2016:
  - Revised throughout to correct the spelling of Picloram (4-amino-3,5,6trichloropicolinic acid
  - Section 7.1.1.3: corrected the preparation instructions for 12N sulfuric acid. The correct volume of water is 200ml (was 300 ml). 100ml concentrated sulfuric diluted into 200 ml water is 12N.

- Revision 9, dated January 06, 2016:
  - Tables 1 and 2: updated sources and concentrations of standards.
  - Section 7.3.1 revised to specify acetone as the diluent.
  - Section 7.3.3.1 (spiking solution prep): updated to reflect current standards and procedures. Sections 7.3.3.2, 7.3.3.3 and 7.3.3.4 deleted (obsolete).
  - Section 7.3.3 (surrogate spiking standard prep): updated to reflect current standards and procedures.
  - Section 8.0: updated minimum sample size (8 oz. glass container).
  - Section 10.1.7 (spiking procedures): updated to reflect current practice and new standard mixes.

- Revision 8, dated December 21, 2011

- Revised the following sections to reflect the requirement that all glassware must be rinsed with sulfuric acid prior to use in the extraction of herbicides: Section 6.2 and Section 10.1.1.
- Added Section 7.1.1.4 which describes the preparation of a 25% sulfuric acid: reagent water solution for use in acid-rinsing glassware.
- Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision

- Revision 7, dated August 26, 2011

  - Section 5.1: revised to include following statement: “Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.”
  - Section 6.2: added EM Quant Peroxide Test Strips to supplies list.
  - Section 7.1: definition and source of Organic-Free Water added.
  - Section 7.1: added requirement to test Ether for peroxide formation.
  - Section 10.1.2: added detail regarding the initial weight of sample to be measured (i.e. ‘Weigh out 30g +/- 0.1g)
  - Revised Section 10.3.4 to include a requirement to pour extract through a funnel plugged with acid washed glass wool. Also revised collection container (was Erlenmeyer flask; now K-D concentrator).
  - Revised Section 10.3.9 to include a procedure for mixing well after adding ethyl ether phase to sodium sulfate.
  - Section 10.6 deleted. Section 10.6 was essentially a duplicate of Section 10.5 that had mistakenly been incorporated into the SOP text.

- Revision 6, dated October 30, 2009

  - Revised SOP format in accordance with Eurofins Corporate Quality SOP No.CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
  - Section 1.1: Expanded to include references to applicable prep and cleanup SOPs.
  - Section 3: revised to reference new location for definitions.
    Section 5: Revised to include most up to date corporate health and safety references and information.
  - Section 7.2.2: Added tables detailing components found in the various standards mixes.
  - Section 7.3: Updated the instructions for preparation of standards.
    Section 7.3: Added tables with calibration standards prep details.
  - Section 8: Updated with additional details including a table outlining containers, preservation and holding times for waters and soils.

- Section 9.1: Expanded QC sample preparation details.
- References: Expanded to include more specific SOP references
  Section 18: Added this Revision History section
- Throughout document: added references to Eurofins LIMS (TALS).

Title: Hot Block Digestion of Sediments, Sludges, and Soils by USEPA Method No(s). SW846 3050B

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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

This SOP covers the acid digestion procedure (SW846 3050B) for the determination of total recoverable elemental constituents in sediments, soils and sludges by inductively coupled plasma emission spectroscopy (EPA Method 6010D) and Inductively Coupled Plasma – Mass Spectrometry (EPA Method 6020B).

1.2. For a complete list of analytes and Reporting limits, refer to the appropriate analytical TestAmerica SOPs: SOP ED-MT-004, *Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy, Method No. SW846 Method 6010D;* SOP ED-MT-029; *Trace Metals Analysis for Water, Wastewater, Soil,Sediment and Leachate Samples by ICP-MS Method SW-846 6020B and* SOP ED-MT-034, *SW846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment, and Leachate Samples by ICPMS*

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A portion of sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the digestate and the sample is refluxed and then brought to a final volume of 100 ml. The resulting digestate is analyzed by ICP-AES or ICP-MS.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences are discussed in the applicable analytical SOPs. Spiked samples are prepared to determine if any interference are present.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the

assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

Hydrogen peroxide (H2O2) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H2O2 to avoid a reaction and possible violent effervescence, or boiling over of the digestion. A splash/splatter hazard is possible and a face shield should be worn.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Block digestor: Environmental Express or equivalent
- AutoBlock: Environmental Express
- Weighing balance capable of accurate weighing to 0.01g.

6.2. Supplies

- watch glasses
- 75 mm funnels
- 50 ml Hot Block Digestion Cups
- Whatman # 41 filter paper or equivalent
- Pipettes varying volumes: Eppendorfs & Fisher
- 4oz Snap Cap Cups from Fisher or equivalent
- Reflux Caps, p/n SC506 Environmental Express or equivalent

7.0 Reagents and Standards

7.1. Reagents

7.1.1. Concentrated Nitric Acid- Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.
7.1.2. Concentrated Hydrochloric Acid- Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.
7.1.3. Reagent Grade Water 18 Megohm Minimum
7.1.4. Hydrogen peroxide (30%), H_2O_2. Store at room temperature; for stability information, refer to manufacturer's instructions.

7.2. Standards -

Storage requirements: all standards are stored at room temperature
Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – 12 months
Working standards – 12 month
(Note: expiration date must not go beyond the expiration date of the source stock).
Concentration: see Attachment 3 for example certificates of analysis (COA) for all of the standards mixes listed below. The COA lists the manufacturer's part number, certified concentration and shelf life.

Standards must be prepared every 12 months or sooner if needed or required. "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

7.2.1. Stock ICP Spike Standards:

- Solutions A, B, C, and D (ICP-Spk), Part No. 4400-100419DD01: commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 1 for the standards certified concentrations and catalog numbers.
- Sulfur 1000 mg/L, single element standard commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions

7.2.2. Working Spike Standard, ICP Working Spike Solution ME_ LCS-int (for ICP prep) - Add the following to a 1000 ml volumetric flask and bring to volume using 5% HNO_3: 50 ml each of Part No. 4400-100419DD01 Solutions A, B, C, and D (Sec 7.2.1.). Record standard preparations in TALS Reagent module. **Note**: Standard must not exceed the earliest expiration date of any one of its components.

7.2.3. Stock ICPMS Spike Standards (Method 6020B)

- ICPMS LCS/SPK: commercially purchased from High-Purity Standards. Store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard doesn't require any pre-dilutions and is ready to be spiked directly into the QC samples. See Attachment 3 for the standards certified concentrations and catalog numbers.

7.2.4. Laboratory Control Sample Reference Material (LCSSRM), Metals in Soil: obtained from Environmental Resource Associates. Store at room temperature; for stability information, refer to manufacturer's instructions.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container[1]	Min. Sample Size	Preservation	Holding Time[2]	Reference
Soils	Glass, plastic	5 grams	Cool 4 ±2°C	6 months	SW846 Method 3050B

[1] All sample containers must be demonstrated to be free of contaminants below the reporting limit.
[2] Holding time is 6 months, however sample analysis should begin as soon as possible.

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with each batch of samples digested or with every Sample Delivery Group (SDG), whichever comes first.

Quality Control	Frequency	Control Limit: Method 6010D & 6020B
Method Blank (MB)	1 in 20 or fewer samples	See the quality control section of the associated analytical SOP
Laboratory Control Sample(LCSSRM)	1 in 20 or fewer samples	Vendor's certified limits
Matrix Duplicate (DU)	1 in 20 or fewer samples	See the quality control section of the associated analytical SOP
Matrix Spike (MS)[1]	1 in 20 or fewer samples	75-125%
Matrix Spike Duplicate (MSD)[1]	When requested by client	See the quality control section of the associated analytical SOP

[1] The sample for DU/MS/MSD is randomly selected, unless specifically requested by a client; predetermined by the prep lab. Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.
[2] Statistical control limits are updated annually and are updated into LIMS.

9.2. Instrument QC

None

10.0 Procedure

10.1. Sample Preparation

10.1.1. All digestion cups and filtration apparatus related to a sample must be labeled with that sample number.

10.1.2. Whenever reflux is required, sample should never be allowed to boil or "bump" as this could result in significant loss of analyte. Sample must never be allowed to go to dryness or be baked.

10.1.3. Mix the sample thoroughly to achieve homogeneity. Weigh 1.00-1.50

grams of sample to the nearest .01 g into a 50 ml hot block digestion cup. QC samples are prepared as follows:

10.1.3.1. Method Blank (MB): Use a blank 50 ml digestion cup for the Method Blank. Attach the TALS (TestAmerica Laboratory Systems) generated label which contains the sample ID number and batch number. The Method Blank is carried through the complete digestion process.

10.1.3.2. Matrix Duplicate (DU): Weigh 1.00-1.50 gm of a duplicate sample to the nearest 0.01 gram into a 50 ml hot block digestion cup. Attach the TALS generated label for the matrix duplicate sample.

10.1.3.3. Matrix Spike Sample (MS): Weigh 1.00 -1.50 gram of duplicate sample to the nearest 0.01 gram into a 50 ml hot block digestion cup. Attach the TALS generated label for the matrix spike sample.

- For ICP preps: Spike 2 ml of ME_LCS-int solution (ICP predigestion spike, Sec 7.2.2) to the digestion cup. If Sulfur is needed, also spike 0.4 mL of Sulfur 1000 mg/L (sec 7.2.1). The resulting elemental amounts are listed in Table 1 (attachment 1). NOTE: the spike must be added prior to the addition of any acids.
- For ICP-MS preps: spike the ICP-MS QC sample with 1.0 ml ICPMS LCS/SPK (Sec 7.2.3). The resulting elemental concentrations are listed in Attachment 2

10.1.3.4. Laboratory Control Sample Reference Material (LCSSRM): Weigh out 1.00-1.50 gm of LCS (sec 7.2.6) to the nearest 0.01 grams into a 50 ml hot block digestion cup. Attach the TALS generated label for the laboratory control sample.

10.1.3.5. Laboratory Control Sample (LCS) for Sulfur:
Using a 50 mL hot block cup, spike 0.4 mL of 1000 mg/L Sulfur (sec 7.2.1).

10.1.3.6. Matrix Spike Duplicate (MSD): when a MSD is requested by the client, prepare the sample in the same manner as the Matrix spike sample (MS), see Matrix Spike Sample prep instructions (Sec 10.1.3.3)..

10.1.4. Using Hot Block (if using AutoBlock, skip to section 10.1.5)

10.1.4.1. Add 10 ml of 1:1 HNO_3, mix the slurry, and cover with a watchglass. Transfer digestion cup to a hot block with the dial setting so that the contents of digestion cup are at

approximately 95°C ±3
°C and reflux for 10 to 15 minutes without boiling or drying the sample.

10.1.4.2. Allow sample to cool, add 5 ml of concentrated HNO_3, replace the watchglass, and reflux for 30 minutes.

10.1.4.3. Repeat step above until no brown fumes are generated.

10.1.4.4. Using a ribbed watchglass, cover the vessel and allow the solution to either evaporate to approximately 5ml or evaporate for 2 hours without boiling. Do not allow sample to go to dryness.

10.1.4.5. After step 10.1.7 has been completed, cool the sample; add 2 ml of deionized water and 3 ml of 30% H_2O_2. Warm cup, and make certain no losses occur due to vigorous effervescence. Heat until effervescence subsides and cool cup.

10.1.4.6. Continue to add 30% H_2O_2 in 1.0 ml aliquots with warming until the effervescence is minimal until the general sample appearance is unchanged. *Note*: Do not add a total of more than 10 ml of 30% H_2O_2.

10.1.4.7. Using a ribbed watchglass, cover the vessel and allow the solution to either evaporate to approximately 5ml or evaporate for 2 hours without boiling. Do not allow sample to go to dryness.

10.1.4.8. Add 10 ml of concentrated HCL to the sample and reflux for 15 min at 95°C ±3.

10.1.4.9. Filter the sample through Whatman 41 filter paper or equivalent into a 4oz Fisher snap cap cup or equivalent. Rinse the 50ml cup using DI water and filter into the 4oz cup. Using deionized water, bring to a final volume of 100 ml. This digestate is now ready for ICP or ICP-MS analyses.

10.1.5. Using AutoBlock

10.1.5.1. Place a Reflux Cap (sec 6.2) on every 50 ml hotblock cup.

10.1.5.2. Turn on AutoBlock then click the *AutoBlock Plus* icon on the computer desktop.

10.1.5.3. Check the waste drum. If needed, appropriately dump the waste.

10.1.5.4. Check the reagents (i.e., HCL, HNO3, 1:1HNO3, 30% H2O2, Deionized Water). Refill if necessary.

10.1.5.5. In the software, click the *Manuel Screen* tab. Enter 95 in the *Temp Control* box. Click *Enable Heat.*

10.1.5.6. Place a 50ml hotblock cup filled with DI water on the autoblock. Cover with a reflux cap. After the hotblock has reached a stable temperature of 95 deg C (~15min), record the temperature using a calibrated thermometer.

10.1.5.7. In the software, click *Exit.*

10.1.5.8. Click *Operator Test* and click *Select Method.*

10.1.5.9. Select *SOILmethod3050B.*

10.1.5.10. Under *Cell Position Selection*, click the hotblock locations where you will have the samples.

10.1.5.11. After closing the see-through plastic door, click on *Start Method.*

10.1.5.12. Don't open the door until the method has been completed.

10.1.5.13. Watch for brown fumes after Nitric Acid has been added by the autoblock. If brown fumes are generated, make note to discard this sample. This sample must be prepared using a Hot Block (sec 10.1.4).

10.1.5.14. When finished, *Method Completed* will be displayed in the software. Record the temp (sec 10.1.5.5).

10.1.5.15. Filter the sample through Whatman 41 filter paper or equivalent into a 4oz Fisher snap cap cup or equivalent. Rinse the 50ml cup using DI water and filter into the 4oz cup. Using deionized water, bring to a final volume of 100 ml. This digestate is now ready for ICP or ICP-MS analyses.

10.2. Calibration

10.2.1. The volume of the 100ml cups are verified for each lot received. The 100 ml volume verification with the appropriate Lot number is documented in the 'Metals labware volume verification logbook.'

10.2.2. Per manufacturer's instructions, recalibrate the AutoBlock every 30 days.

10.2.2.1. Place an empty hotblock cup on an analytical balance and 'zero' the balance.
10.2.2.2. Place that empty hotblock cup in the first position, I1, in the AutoBlock.
10.2.2.3. Click the *AutoBlock Plus* icon on the computer desktop
10.2.2.4. Click the *Manuel Screen* tab
10.2.2.5. Under *Reagent Selection*, click *H2O(6).* This is Port 6 which contains Deionized Water
10.2.2.6. Under *Cell Position Selection,* click *I1*
10.2.2.7. Under *System Control*, click *Move to Position*
10.2.2.8. In the *Injection Amount (ml)* box, type "30" and press *Inject.* The Autoblock will dispense 30 mL of DI Water into the cup.
10.2.2.9. Click *Move to Drain* and press *Exit*
10.2.2.10. Weigh the cup containing the DI Water on the zero'd balance to determine the weight of the water
10.2.2.11. Press the *Service Screen* tab
10.2.2.12. Press the *Pump Cal* tab
10.2.2.13. Under *Pump 2 Actual Injection Amount (ml)*, enter the weight of the water and press *Pump 2 Save Cal.*
10.2.2.14. Document the calibration on the Autoblock maintenance logbook.

10.3. Sample Analysis

Refer to the appropriate Analytical SOPs listed in Sec 1.2.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration :

Refer to appropriate Analytical SOPs listed in Sec 1.2.

11.4. Documenting and Reporting

11.4.1. Sample preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS).

11.4.1.1. Double click the TALS icon on the computer desktop. Enter your username and password and select Login.

11.4.1.2. Under TALS Menu, click the plus sign ('+') next to Analyst. Double click Analyst Desktop II. Click the plus sign ('+') next to Methods.

11.4.1.3. Right click the prep method that you are using to prep the samples. Select 'Create New Batch' then select 'From Scratch.'

11.4.1.4. The analyst must enter the following information: Sample names (use scanners), initial and final sample volume, spike name and amount used, and all reagents and their corresponding lot numbers.

11.4.1.5. After saving the batch information in TALS, select Print-Batch Sheets (which must be submitted to metals analytical dept. along with the samples) and select Print-Batch Labels (which must be attached to the corresponding final digestate sample cups in the batch).

11.4.2. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable preparation batch.

11.4.2.1. Open the preparation batch and click on the Edit tab above to enter the Edit Mode.

11.4.2.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.4.2.3. Acknowledge by filling in responses to all unacknowledged findings.

11.4.2.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.4.2.3.2. Fill in the appropriate comments in the response box, then hit 'OK.'

11.4.2.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the

primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; SW-846, Method 3050B.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-MT-004, Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy via SW846 Method 6010D, most current revision.

15.6. TestAmerica Edison SOP No ED-MT-029, Trace Metals Analysis for Water, Wastewater, Soil,Sediment and Leachate Samples by ICP-MS Method SW-846 6020B, most current revision.

15.7. TestAmerica Edison SOP ED-MT-034, *SW846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment, and Leachate Samples by ICPMS*, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Table 1, Matrix Spike concentration for Soil samples by 3050B-ICP
Attachment 2: Table 2, Matrix Spike concentration for Soil samples by 3050B-ICPMS
Attachment 3: Certificate of analysis of each stock standard.

18.0. Revision History

- Revision 15, dated 29 September 2021
  - Sec. 10.1.3.3: Revised the amount of ICPMS LCS/SPK added for ICP-MS preps to 1.0 ml to reflect actual laboratory practices.

- Revision 14, dated 30 December 2019
  - Sec 6.1: Added AutoBlock to the instrument list.
  - Sec 6.2: Added Reflux Caps to the supplies list
  - Sec 7.2.1: Added Sulfur to the stock ICP spike standards list.
  - Sec 10.1.3.3: Added matrix spiking instructions for Sulfur.
  - Sec 10.1.3.5: Added LCS preparation instructions for Sulfur.
  - Sec 10.1.5: Added instructions for using the AutoBlock.
  - Sec 10.2.1: Replaced the 50ml volume verification with a 100ml volume verification.
  - Sec 10.2.2: Added procedure for the monthly recalibration (volume dispenser) of the Autoblock.
  - Table 1: Added spike amount for Sulfur.

- Revision 13, dated 05 August 2019
  - Updated SOP header with Eurofins emblem.
  - Throughout the SOP deleted all references to Methods 6010B, 6010C, 6020 and 6020A.
  - Sec 2.0: Revised the final volume of digestates from 50ml to 100ml to reflect actual lab practices.
  - Sec 6.2: Added 4oz snap seal cups to the list of supplies.
  - Sec 10.1.3.3: Revised the spiking volume to add for ICP-MS LCS from 1ml to 2ml.
  - Sec 10.1.12: Clarified instructions for filtering digestates; added final volume of digestates.

- Revision 12, dated 06 September 2018
  - Throughout the SOP - revised to reference the analytical methods 6010D and 6020B.
  - Sec 11.4.: Updated instructions for documenting and reporting preparation batch information in TALS and instruction for using TALS Data Review Checker (DRC) program.

- Revision 11, dated 13 July 2016

  - Sec 1.1: Added method 6010C and 6020A
  - Sec 1.2: Deleted reference to SOP ED-MT-033; SOP has been combined with SOP ED-MT-004.
  - Sec 9.1: Added MSD to the list of Sample QC including the frequency and control limit requirements.
  - Sec 10.1.3.5: Added preparation instructions for MSD.
  - Sec 15.7: Removed reference to SOP No ED-MT-033, subsequent section adjusted accordingly.

- Revision 10, dated 05 June 2014

  - Sec 1.2: Removed SOP reference ED-MT-031 and added SOP reference ED-MT-033 and ED-MT-034.
  - Sec 2.0: Corrected typo error for the final volume from 100 ml to 50 ml as described in this SOP.
  - Sec 6.2: Removed 125 ml specimen cups.
  - Sec 10.1.1: Deleted the use of glassware – not applicable for this SOP.
  - Sec 15.7 & 15.8: Added TestAmerica Edison SOP No. ED-MT-033 and ED-MT-034 to the list of reference.
  - Attachment 1: Added Boron to the ICP matrix spike list.
  - Attachment 2: Added Zinc to the ICPMS matrix spike list and deleted Mercury from this list; corrected Boron's matrix spike concentration.

- Revision 9, dated 31 May 2012

  - Sec 1.3 and 12: Revised LQM references to reflect the most current LQM revision.
  - Throughout the document: Removed all references to CLP SOW ISM 01.2
  - Sec 6.1: added 'or equivalent'.
  - Sec 7.2.1: Updated the ICP stock standard to reflect current lab practices.
  - Sec 7.2.2: Updated the preparation of the working spike standard.
  - Sec 7.2.3: Replaced the vendor for the ICPMS stock standards to High-Purity Standards (former vendor- CPI International).
  - Deleted Sec 7.2.4 & 7.2.5 (CLP ISM01.2 Standards, not applicable); subsequent sections adjusted accordingly.
  - Sec 9.1 Table: MB and DU control limits have been updated and now reference the associated analytical SOP for the most current QC limits.
  - Sec 10.1.3.1 – 10.1.3.4: Updated the digestion cup labeling procedure using the TALS generated labels
  - Sec 10.1.12: Revised procedure to reflect actual lab practices - *samples are brought to a 50ml final volume and then filtered.* Final digestate volume revised from 100 ml to 50 ml.
  - Sec 10.2.1: Replaced the 125 ml hot-block cups with 50 ml size cups
  - Sec 13 & 14: Updated to comply with TestAmerica Corporate Quality SOP No. CW-QS-002, Writing a Standard Operating Procedure (SOP), most current revision.
  - Attachment 3: Replaced COAs to reflect the updated stock standards.

- Revision 8, dated 30 March 2010

  - Sec 1.1: Deleted Table which listed the 6010B RLs for soils; added Sec 1.2 to include list of analytical SOPs where the appropriate RLs can be found.
  - Sec 7: Added matrix spike and LCS standards for Method 6020 and ISM01.2
  - Sec 8: Added the holding time for soil samples analyzed by Method CLP SOW ISM01.2.
  - Sec 9.1: Added control limits for CLP SOW ISM01.2.; Deleted Blank spike and LCS duplicate.
  - Sec 10: Revised amount of sample to be weighed to 1.00-1.50 grams as per CLP Method ISM01.2
  - Sec 10: Revised hot block temperature to 95± 3 deg, to follow the more stringent setting.
  - Deleted section previously identified 10.1.3.2, Blank spike; subsequent sections adjusted.
  - Sec 10.1.3.3 & Sec 10.1.3.4: Revised to include spike procedures for ICP and ICPMS, Method 6020 and ISM01.2.
  - Sec 10.1.3.5: Deleted LCS duplicate
  - Sec 10.1.9: Section added to reflect actual lab practices; subsequent section numbers adjusted.
  - Sec 10.1.10: Text added: '*Note: For samples which are being digested for CLP SOW ISM01.2 ICP-MS, do not perform this step.'*
  - Sec 15: Added applicable references.
  - Sec 17: Added attachments 2 & 3
  - Attachment 3: Added applicable COAs
  - Added this revision history.

- Revision 7, dated June 13, 2008
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1: Added laboratory's reporting limits in Table format.
  - Section 7 Reagents & Standards: Included storage and stability information of reagents and standards. Updated the source of the stock spike standards and the preparation instructions for the working spike standards.
  - Removed all reference related to GFAA analysis including standard solutions, digestion procedure and Table 2: Spike amount for soil samples by GFAA; clarified the digestion procedure for ICP-AES analysis.
  - Section 10.1 Sample Preparation: Deleted acid rinsing of glassware since 50 ml digestion cups are being used for digesting samples.
  - Section 10.1 Sample Preparation: Replaced hot plate setting of 2.5 to hot block setting of 95°C.
  - Section 15 References: Added applicable references

Attachment 1

TABLE 1

Matrix Spike Concentration for Soil Samples by ICP	
ELEMENT	SPIKE AMOUNT (mg/Kg)
Aluminum	200
Antimony	50
Arsenic	200
Barium	200
Beryllium	5.0
Cadmium	5.0
Boron	50
Calcium	2000
Chromium	20
Cobalt	50
Copper	25
Iron	100
Lead	50
Manganese	50
Magnesium	2000
Molybdenum	50
Nickel	50
Potassium	2000
Selenium	200
Silver	5.0
Sodium	2000
Strontium	50
Thallium	200
Tin	50
Titanium	50
Vanadium	50
Zinc	50
Sulfur	400

Matrix spike sample concentration: Dry weight correction is made at final calculation

Attachment 2

ICPMS Matrix Spike Concentration Method 6020	
Element	**Matrix Spike Conc. (mg/Kg)**
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	100
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	50

Note: For Matrix spike sample concentration, dry weight correction is made at final calculation.

Attachment 3

ERA
A Waters Company

Certificate of Analysis

Lot No. D075-540

Metals in Soil

Catalog No. 540
Issue Date: October 11, 2011
Revision Date: Original

Certification

Parameter	Total Concentration [1] (mg/kg)	Certified Value [2] (mg/kg)	Uncertainty [3]	QC PALs™ [4] (mg/kg)	PT PALs™ [5] (mg/kg)
aluminum	68100*	10100	0.8%	5110 - 15100	4560 - 15600
antimony	228	113	0.8%	DL - 252	22.7 - 286
arsenic	266	237	2.9%	197 - 278	169 - 306
barium	678	252	2.4%	212 - 292	186 - 318
beryllium	105	93.3	4.1%	77.7 - 109	69.4 - 117
boron	135	118	1.3%	87.3 - 149	71.9 - 164
cadmium	215	191	4.5%	159 - 223	140 - 242
calcium	25400*	6840	5.3%	5640 - 8040	5080 - 8600
chromium	364	128	3.8%	104 - 151	89.3 - 166
cobalt	208	178	3.5%	149 - 207	132 - 223
copper	145	123	9.0%	103 - 143	92.3 - 154
iron	42900*	13100	4.8%	6650 - 19600	4290 - 22000
lead	105	103	6.7%	85.6 - 120	73.0 - 132
magnesium	9060	2990	4.4%	2280 - 3710	1980 - 4010
manganese	918	333	5.5%	274 - 391	250 - 415
mercury	12.8	12.4	10.1%	8.88 - 15.9	6.36 - 18.4
molybdenum	113	91.1	4.8%	74.2 - 108	63.3 - 122
nickel	152	118	6.0%	96.8 - 139	85.8 - 150
potassium	19700*	2870	4.0%	1950 - 3780	1780 - 3950
selenium	123	110	1.6%	86.6 - 134	72.6 - 148
silver	51.6	47.3	3.8%	31.3 - 63.2	31.4 - 63.2
sodium	17400*	550	3.8%	405 - 694	289 - 811
strontium	252	128	4.0%	104 - 153	90.6 - 166
thallium	177	158	3.7%	128 - 188	108 - 208
tin	121	107	0.8%	84.5 - 130	61.7 - 153
titanium	3280*	282	4.8%	65.4 - 498	0.00 - 606
vanadium	205	119	4.0%	94.9 - 143	81.2 - 157
zinc	197	183	6.9%	150 - 216	127 - 239

Please see footnotes on back

DL = Detection Limit

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SOLN. A
DE
4-4-11

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Recd
03/22/11 MP

TestAmerica/Edison
Custom Standard
5% HNO3

Concentrations in ug/mL ± 0.5%

Fe	1000	Zn	500
Se	2000	Mn	500
TL	2000	Ni	500
Be	50		
Cd	50		
Cr	200		
Co	500		
Cu	250		
Pb	500		
Ag	50		
Sr	500		
V	500		

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution B**
Lot Number: **11C157**
Shelf Life: **18 Months**

TestAmerica/Edison
Custom Standard
10% HNO3

Date Recd
03/22/11 MP

Concentrations in ug/mL ± 0.5%

Al	2000
Mg	20000
K	20000
Ca	20000
Na	20000

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution C**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
03/22/11 MP

TestAmerica/Edison
Custom Standard
5% HNO3 + 2% HF

Concentrations in ug/mL ± 0.5%

Sb	500
Mo	500
Sn	500
Ti	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution D**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
05/22/11 MP

TestAmerica/Edison
Custom Standard
2% HNO3

Concentrations in ug/mL ± 0.5%

As	2000
Ba	2000
B	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

SM-606-111
(ICPMS LCS/SPK)
Element List
(µg/mL)

Aluminum	500
Antimony	5
Arsenic	10
Barium	10
Beryllium	5
Boron	100
Cadmium	5
Calcium	500
Chromium	10
Cobalt	5
Copper	10
Iron	500
Lead	5
Magnesium	500
Manganese	50
Molybdenum	10
Nickel	10
Potassium	500
Selenium	10
Silver	5
Sodium	500
Strontium	10
Thallium	4
Tin	10
Titanium	10
Vanadium	10
Zinc	50

Title: Procedure for the Microwave Extraction of Solids, SW846 3546

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Sylvanus Klusey Organic Operations Manager	3/12/18 Date	Dan Helfrich Health & Safety Manager	3/12/18 Date
Carl Armbruster Quality Assurance Manager	3/12/18 Date	Mark Acierno Laboratory Director	3/12/18 Date

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1.0 Scope and Application

The procedure uses microwave energy to produce elevated temperature and pressure conditions (i.e., 100° -115°C and 50 - 175 psi) in a closed vessel containing the sample (soil, sediment and sludge) and organic solvent(s) to achieve analyte recoveries equivalent to those from Automated Soxhlet extraction (Method 3541), using less solvent and taking significantly less time than the Automated Soxhlet procedure.

This method is applicable to the extraction of semi-volatile petroleum products in soil, sediment and sludge which may then be analyzed by a variety of chromatographic procedures. The method may also be applicable for the extraction of additional target analytes, provided that the analyst demonstrates adequate performance for the intended application as further described in TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

This method only is applicable to solid samples with small particle sizes. If practical the sample may be air dried and ground to a fine powder prior to extraction.

1.1 **Analytes, Matrix(s), and Reporting Limits:** This standard operating procedure details the procedures used by TestAmerica Edison for the microwave extraction of soils, sediments, sludges, and waste solids for semivolatile organic compounds by SW846 Method 3546 for subsequent analysis of:

- Diesel Range Organics (C10-C28 & C8-C44) by SW846 Methods 8015B, 8015C and 8015D (see TestAmerica Edison SOP Nos. ED-GCS-009 and ED-GCS-020);
- TPH by NJDEP-OQA-QAM-025 (see TestAmerica Edison SOP No. ED-GCS-011);
- Organochlorine Pesticides and PCBs by SW846 8081A/B and 8082/A (see TestAmerica Edison SOPs No.ED-GCS-003, ED-GCS-004, ED-GCS-016 and ED-GCS-017)
- Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS) by SW846 8270 and 8270D (TestAmerica Edison SOPs ED-MSS-002 and ED-MSS-009)
- PCBs by GC/MS using EPA Method 680 (TestAmerica Edison SOP ED-MSS-010)

1.2 The microwave prep for NJDEP Extractable Petroleum Hydrocarbons (EPH) (10/08), current revision, is addressed separately in TestAmerica Edison SOP ED-GCS-012 (current revision).

1.3 For a complete discussion of analytes and reporting limits (RLs) please refer to the applicable TestAmerica Edison analytical SOPs referenced in Section 1.1 and in References (Section 14.0).

1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A representative soil, clay, sediment, sludge, solid or waste sample is weighed and placed into a 75mL Teflon vessel and extracted using an appropriate solvent in a microwave extractor. The extraction vessel containing the sample and solvent system is heated to the extraction temperature and extracted for 10 minutes. The mixture is allowed to cool. The vessel is opened and the contents are filtered. The solid material is rinsed and the various solvent fractions are combined. The extract is then dried, concentrated using a nitrogen blow down device, and as necessary, solvent exchanged for use in clean up, fractionation, or determinative methods.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may contribute to interferences with sample analysis. All materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing a method blanks.

4.2 Phthalate esters are commonly extracted from laboratory items. Plastics in particular, must be avoided because phthalates are easily extracted from these materials. The presence of these contaminants in extracts is problematic because it will give false positive results for the methods that list phthalates as target compounds or it will interfere with the target compounds of other methods that are co-extracted with them.

4.3 All glassware used must be scrupulously cleaned before use in trace analysis. Refer to TestAmerica Edison SOP No. ED-GEN-013 (*Glassware Cleaning*), current revision.

4.4 Use caution if sodium sulfate is used for this method. Salts are known to super heat when used in microwave. Typically samples are analyzed with the addition of 5.0 grams sodium sulfate.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The use of the microwave to extract samples creates excessive pressure and temperature within the digestion vessels very rapidly. Cooling the sample after digestion is extremely important.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Zymark Turbovap II concentrator
- Electronic Balance, capable of weighing to 0.01g.
- N-Evap Analytical Evaporator
- Fisher 8 Position Steam Bath
- MARS 5 microwave oven with temperature sensor (CEM Corp) capable of sensing

temperature within 2.5°C and adjusting microwave field output within 2 sec of sensing.
- MARS 40 position carousel (CEM Corp)
- Visiprep DL column holder

6.2 Supplies

- 10, 50 and 200 mL Concentrator tubes.
- Vials, - 1ml, 4ml, and 10ml with PTFE lined snapcaps.
- Glass syringes, 25ul, 50ul, 100ul, 500ul, 1000ul
- Pastuer pipets
- Glass Buchner funnels (stainless steel funnels may be substituted) – Fisher Scientific.
- 500 ml Kuderna-Danish (KD) flask
- Stainless steal spatulas
- Glass wool- Contaminant-free (silane treated or oven baked at 400°C)
- 75mL Teflon Express vessels with stopper and cap (CEM Corp.)
- Glass fiber filter paper - #1 185mm (Whatman)
- Weigh boats
- Tongue depressors, wood

7.0 Reagents and Standards

Note: Each solvent lot is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.1. Reagents

7.1.1 Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At TestAmerica Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).

7.1.2 Methylene Chloride, JT Baker Resi-analyzed, P/N 9254-03 (or equivalent)

7.1.3 Hexane – JT Baker Ultra-Resi 73513-42-5 or equivalent

7.1.4 Acetone, J.T. Baker Ultra- ResiAnalyzed, Catalog No. 9264-03 (or equivalent)

7.1.5 Nitrogen, high-purity

7.1.6 Construction Sand (Home Depot or Lowes), free from organic compounds. (Baked in the muffle furnace for four hours at 400°C and stored in a dessicator prior to use.)

7.1.7 Sodium Sulfate Anhydrous Powder, Mallinckrodt MA8020-06 or equivalent (Must be baked in the muffle furnace for four hours at 400°C and stored in a dessicator prior to use.)

7.2. Standards

7.2.1 Stock target analyte standard spiking solutions are purchased as prepared solutions (see table below). Stock solutions are diluted (in volumetric glassware) to a working concentration using methylene chloride ($MeCl_2$) hexane or Acetone as the diluent as indicated below below. Stock standards of similar quality from other suppliers may be substituted as required.

NOTE: Second sources (from separate lots) are used for quanitation standards and spiking.

Stock Standards (Methods 8015B,8015C and 8015D DRO & NJDEP OQA-QAM-025)				
Standard Mix	**Concentration**	**Source**	**Catalog #**	**Used in method(s):**
TPH Mix 3	1000 ppm	Sigma Aldrich	861394U	*NJDEP OQA-QAM-025
o-Terphenyl	10000 ppm	Sigma Aldrich	47580U	*SW846 8015B, 8015C &8015D DRO *NJDEP OQA-QAM-025
Chlorobenzene	10000 ppm	Sigma Aldrich	4 6860U	*NJ DEP OQA-QAM-025
Diesel Fuel #2	50000 ppm	Restek	31259	*SW846 8015B, 8015C &8015D DRO *NJDEP OQA-QAM-025

Stock Standards - Pesticides/PCBs* (Methods SW846 8081A/B and 8082/A)				
Standard Mix	**Concentration**	**Source**	**Catalog #**	**Used in method(s):**
Pesticide Surrogate Spike Mix ("Pest/PCB Surrogate")	10 ug/ml	Supelco	861275	SW846 8081/8082
Aroclor Spike Mix ("PCB Spike")	100 ug/ml	Supelco	861274	SW846 8082
SS CLP Organochlorine Pesticides Mix ("Pest Spike")	2000 ug/ml	Supelco	4S7426-U	SW846 8081
Chlordane (technical) ("Technical Chlordane Spike")	1000 ug/ml	Supelco	48065-U	SW846 8081
Toxaphene	1000 ug/ml	Supelco	48065-U	SW846 8081

*May be substituted with equivalent standards from alternate sources.

Components of Pesticide/PCB Standard Mixes (Methods SW846 8081A/B and 8082/A)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Decachlorobiphenyl (DCB)	Supelco – 4S8913	Pest/PCB Surrogate	10.0
2,4,5,6-Tetrachloro-m-xylene (TCMX)	Supelco – 4S8913	Pest/PCB Surrogate	10.0
Aroclor 1016	Supelco - 861274	PCB Spike	100

Components of Pesticide/PCB Standard Mixes (Methods SW846 8081A/B and 8082/A)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Aroclor 1260	Supelco - 861274	PCB Spike	100
Aldrin	Supelco - 4S7426-U	Pest Spike	2000
Alpha-BHC	Supelco - 4S7426-U	Pest Spike	2000
Alpha-Chlordane	Supelco - 4S7426-U	Pest Spike	2000
Beta-BHC	Supelco - 4S7426-U	Pest Spike	2000
Delta-BHC	Supelco - 4S7426-U	Pest Spike	2000
Dieldrin	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan I (Alpha)	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan II (Beta)	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan Sulfate	Supelco - 4S7426-U	Pest Spike	2000
Endrin Aldehyde	Supelco - 4S7426-U	Pest Spike	2000
Endrin Ketone	Supelco - 4S7426-U	Pest Spike	2000
Endrin	Supelco - 4S7426-U	Pest Spike	2000
Gamma-BHC (Lindane)	Supelco - 4S7426-U	Pest Spike	2000
Gamma-Chlordane	Supelco - 4S7426-U	Pest Spike	2000
Heptachlor	Supelco - 4S7426-U	Pest Spike	2000
Heptachlor Epoxide (Isomer B)	Supelco - 4S7426-U	Pest Spike	2000
Methoxychlor	Supelco - 4S7426-U	Pest Spike	2000
4,4’-DDD	Supelco - 4S7426-U	Pest Spike	2000
4,4’DDE	Supelco - 4S7426-U	Pest Spike	2000
4,4’-DDT	Supelco - 4S7426-U	Pest Spike	2000
Chlordane (technical)	Supleco – 48065-U	Technical Chlordane Spike	1000
Toxaphene	Supelco 48103	Toxaphene Spike	1000

Stock Standards – BNAs (Method 8270x)			
Standard Mix	**Concentration (ug/ml)**	**Lab Vendor**	**Catalog #**
8270 List 1/ Std#1 MegaMix	500/1000/2000	RESTEK	567672
8270 List 1/ Std#5 N-Nitrosodiphenylamine	2000	RESTEK	567676
8270 List 1/ Std#2 Amines	2000	RESTEK	567673
8270 List 1/ Std#3 Benzoic Acid	2000	RESTEK	567674
Custom SVO Mix	2000	SPEX	SVO-TANJ-16-5
Bisphenol A	1000	SPEX	S-509-MC
8270 List 1/ Std#8	2000	RESTEK	568724
8270 Surrogate Standard	5000	RESTEK	567685
Aromatic Amines Custom Mix	2000	Supelco	21467482

Stock Standards – PCB Homologues (Method 680)			
Standard Mix	**Concentration (ug/ml)**	**Lab Vendor**	**Catalog #**
Retention Time Calibration Standard Mixture	Varies by component (100 to 200)	Ultra	CB-682MN
Concentration Calibration Standard Mix	Varies by component (50 to 250)	Ultra	CB-681MN
13C12-Decachlorobiphenyl Surrogate	40	Cambridge	EC-1410-3
Lindane13C6	100	Cambridge	CLM-1282-S

Components of PCB Homologue Mixes (Methods 680)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
PCB-104	Ultra CB-682MN	Retention Time Calibration Standard Mixture	100
PCB-208	Ultra CB-682MN	Retention Time Calibration Standard Mixture	200
PCB-77	Ultra CB-682MN	Retention Time Calibration Standard Mixture	100
Total Nonachlorobiphenyls	Ultra CB-682MN	Retention Time Calibration Standard Mixture	200
DCB Decachlorobiphenyl	Ultra CB-681MN	Concentration Calibration Standard Mix	250
Total Dichlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50
Total Heptachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	150
Total Hexachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Monochlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50
Total Octachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	150
Total Pentachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Tetrachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Trichlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50

7.2.2 Spiking Standards Prep for GC FID (DRO/QAM): The table below provides instruction in preparation of the various working spiking standards for use in this preparation method using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with the indicated solvent.

Working Spiking Standards Preparation					
Standard Name	**Vendor/ Cat #**	**Initial Conc. (ug/ml)**	**Vol. of Standard (ml)**	**Vol. of solvent mls (solvent)**	**Final Conc. (ug/ml)**
DRO Spike/ QAM-025 LCS Spike Diesel Fuel #2	Restek/31259	50000	4	100 ($MeCl_2$)	2000
DRO Surrogate -o-terphenyl	Supelco/47580U	10000	1.0	500 (Acetone)	20
QAM-025 MS/MSD Spike TPH Mix 3	Sigma/861394-U	1000	1.47	10 ($MeCl_2$)	147
QAM-025 Surrogate -Chlorobenzene -o-terphenyl	Supelco/46860-U Supelco/47580-U	10000 10000	1.0 1.0	500 500	20 20

7.2.3 Spiking Standards Prep for GC ECD(8081/8082):

7.2.3.1 All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware. **Note:** septa on all surrogate and spike vials are to be replaced immediately after use. Additionally, all surrogate and spike vials are to be returned to the standards refrigerator immediately after use.

7.2.3.2 Pesticide Spiking Standard (LCS/MS/MSD): The Pesticide Mix BS/MS/MSD solution with 20 single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul (0.50 ml) of the 2000 ppm Pest Spike stock solution (Supelco 4S7426-U, see tables in Section 7.2.1) to a 50 ml final volume with acetone. For spiking instructions refer to Section 10.

7.2.3.3 PCB Spiking Standard (LCS/MS/MSD): The PCB spiking solution (Supelco – 861274, see Tables 1 and 2) is a custom mix received from the vendor at its working concentration of 100 ug/ml in Acetone. For spiking instructions refer to Section 10.

7.2.3.4 Technical Chlordane Spiking Standard (LCS/MS/MSD): A technical Chlordane spiking solution is prepared at a final concentration of 100 ug/ml by diluting 1 ml of the 1000 ug/ml Technical Chlordane Spike solution (Supelco 48065-U, see tables in Section 7.2.1) to a 10 ml final volume using acetone. For spiking instructions refer to Section 10.

7.2.3.5 Toxaphene Spiking Standard (LCS/MS/MSD): A Toxaphene spiking solution is prepared at a final concentration of 100 ug/ml by diluting 1 ml of the 1000 ug/ml Toxaphene Spike solution (Supelco 48103, see tables in

Section 7.2.1) to a final volume 10 mL.using acetone. For spiking instructions refer to Section 10.

7.2.3.6 Pesticide/PCB Surrogate Spiking Standard: The Pesticide/PCB surrogate spiking solution is a concentrated 10 ml solution of DCB/TCMX at a concentration of 10 ug/ml. (Supelco Catalog # 861275, see tables in Section 7.2.1). The concentrate is used without further dilution prior to the spiking of samples.

7.2.4 Spiking Standard for BNA: For use in spiking Soils samples prepared for BNA analysis by SW846 Method 8270. Prepare the spiking solution for MS/MSD/Blank Spike (LCS) as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	20 ml (methanol)	50/100/200 ppm
8270 List 1/ Std#5 N-Nitrosodiphenylamine	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/ Std#2 Amines	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/ Std#3 Benzoic Acid	2000ppm	10 ml (methanol)	100 ppm
Custom SVO Mix	2000ppm	10 ml (methanol)	100 ppm
Bisphenol A	1000ppm	10ml (methanol)	50ppm
8270 List 1/ Std#8	2000ppm	20ul (methanol) 100ul(methanol)	Used neat for LVI Used neat for non-LVI
Aromatic Amines Custom Mix**	2000ppm	10 ml (methylene chloride)	100 ppm

** *As needed based on clients requirement.*

7.2.4.1 BNA Surrogate Standard Spiking Solution: For use in spiking all blanks, samples and associated QC prior to extraction. Prepare as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 1000 ml methanol	Final Concentration
8270 Surrogate Standard	5000 ppm	20 ml	100 m

7.2.5 Spiking Standard for PCB Homologues (EPA 680): For use in spiking LCS and MS/MSD prior to extraction. Prepare by adding 1.0ml

of the Retention Time Calibration Standard and 1.0ml of the Concentration Calibration Standard Mix to hexane in volumetric glassware and dilute to final volume of 100ml. 1.0mL of this solution is spiked into LCS/MS/MSD prior to extraction.

7.2.5.1 PCB Homologue Surrogate Standard Spiking Solution (EPA 680): (2.0ug/mL and 2.5ug/mL) – Add 3.125mL of $^{13}C_{12}$-Decachlorobiphenyl and 1.0mL of Lindane13C6 and dilute to a final volume of 50mL in hexane in volumetric glassware. 1.0mL of this solution is spiked into all blanks, samples and QC items prior to extraction.

7.2.6 The preparation of all standards must be documented in a standard preparation logbook or in the TALS reagent module. Information such as standard supplier, lot number, original concentration, and a description of how standard was prepared are required along with a laboratory lot number, analyst's initials, date prepared and verification signature. Standards must be made every 6 months or sooner if signs of degradation appear.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Glass	4 oz	Cool 4 ± 2°C	14 Days	a) NJDEP-OQA-QAM-025; b)SW846 Method 8000

Samples are collected in amber jars with Teflon lined caps.
[1]All sample extracts must be analyzed within forty (40) days of extraction.

9.0 Quality Control

9.1. Sample QC – The following quality control samples are prepared with each batch of samples.

QC Analysis	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS/LCSD)[1]	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria
Method Blank (MB)	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria

QC Analysis	Frequency	Acceptance Criteria	Corrective Action
MS\MSD[2] (Client specified)	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria
Surrogates[3]	All Samples including QC	See applicable analytical SOPs	See applicable analytical SOPs for control criteria

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract except for NJDEP EPH 10/09 where LCS/LCSD is required.
[2] The sample selections for MS/MSD are randomly selected, unless specifically requested by a client. For NJDEP EPH 10/09, only MS is required (MSD for NJDEP EPH 10/09 is by client request only).
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.2. Instrument QC

N/A

10.0 Procedure

10.1. Sample Preparation

10.1.1 Decant and discard any water layer on a sediment sample. Discard any foreign objects such as rocks, leaves and sticks. Mix sample thoroughly with wooden tongue depressor prior to subsampling

10.1.2 The initial amount and treatment of soil samples is method dependent:

- For QAM-025, DRO (8015): 15 grams of soil is weighed into a 75mL Teflon microwave vessel (the weight is recorded to two significant figures). Where practical samples should be air dried and ground prior to extraction. Drying should be performed in a hood to avoid contamination.
- For 8270/ 8081/8082: 15 grams of soil is weighed into an aluminum weighing dish (the weight is recorded to two significant figures). 15 mls of acetone is added to the dish and the soil/acetone mixture is stirred well with a wooden tongue depressor to create a slurry. The soil/acetone slurry is transferred to a 75 ml Teflon microwave vessel. The dish is then rinsed with 15 ml of methylene chloride which is also added to the microwave vessel.
- For EPA 680: 30 grams of soil is weighed into a 75mL Teflon microwave vessel (the weight is recorded to two significant figures).

10.1.3 For laboratory blanks and control spikes, an equal portion of clean sand is prepped along with samples.

10.1.4 All samples, blanks, and spikes are fortified with an appropriate amount of the applicable method surrogates. All matrix spikes and lab

control spikes are fortified with an appropriate amount of the applicable method spiking solution. See Attachment 1 for general spiking instructions. (Refer to Section 7.2 for information on preparation of spiking solutions):

- PCBs (8082): add 50 ul of the PCB Spike solution to each LCS and designated MS/MSD;
- Pesticides (total, 8081): 100 ul of the Pest Spike solution to each LCS and designated MS/MSD;
- Technical Chlordane: 100 ul of the Technical Chlordane Spike mix to each LCS and designated MS/MDS;
- Toxaphene: 100 ul of the Toxaphene Spike mix to each LCS and designated MS/MSD;
- BNA (8270): 500ul of spike/Surrogate solution to each LCS and designated MS/MSD);
- PCB Homolgues (EPA 680): 1.0 ml each of Spiking solution (see Sec.7.2.5) and Surrogate solution (see Sec. 7.2.5.1) to each LCS and designated MS/MSD.

NOTE: When spiking the samples, it is critical to remove all bubbles from the syringe.

10.1.5 Add the following solvent to each sample vessel depending upon the fraction (**note**: for Pesticides and PCBs by methods 8081 and 8082 the solvent addition is addressed in Section 10.1.2 above)

- 30mL of methylene chloride for DRO (8015)/QAM-025;
- 30 mL of Methylene Chloride/Acetone blend (50:50) for BNA (8270)

30 mL of Hexane for PCB homologues by EPA 680

10.1.6 A stopper is placed over the opening along with the vessel cap. The vessel is then tightened using the MARS capping wrench. This capping wrench tightens the cap to the appropriate torque.

10.1.7 The sample vessels are placed into the 40-position carousel. Each position has an insulator sleeve that the vessel slides into – slide the vessel into the appropriate sleeve – and press firmly down to ensure a complete connection.

10.1.8 Place the carousel (with samples and vessels) into the microwave unit.

Start Run: (load method – using key pad on face of unit)
Press: Home

Press Select: load method
Press Select: from user directory
Press Select: 3546 Microwave – Xpress method
Press Start.

Microwave Operating Parameters				
Power Max	**Power %**	**Oven Ramp**	**Degrees Celsius**	**Hold Time**
1200W	100	10.0 minutes	110	10.0 minutes
1600W[1]	100	20.0 minutes	115	10.0 minutes

[1] For pesticide/PCB and BNA samples only

10.1.9 A 5-minute cool down will close out the run.

10.1.10 Remove vessels from carousel and allow cooling (either at room temperature or in a cold water bath).

10.1.11 When cool down is complete, remove carousel from microwave.

10.1.12 Pre-wash a funnel filled with sodium sulfate with three successive washings of 30 ml acetone. Pour the extract carefully through the sodium sulfate funnel (to remove any moisture) into the 200mL concentrator tube. Rinse the 75mL vessel with 10 mL methylene chloride three times to ensure quantitative transfer.

10.1.13 Set the water temperature in the concentrator to 35°C and adjust the pressure to around 5psi of nitrogen.

10.1.14 Place the tube into the apparatus and slowly raise the pressure to 20psi. If splashing occurs, start with a lower pressure and raise it when the solvent gets lower.

10.1.15 Concentrate to just under the 1mL mark on the nipple of the tube. Remove from the bath and transfer to an auto sampler vial with a glass Pasteur pipette..

10.1.16 Add a few drops of clean methylene chloride to the tube, rinsing the narrow part and add to the vial to adjust to 1mL or other method specific final volume. If solvent exchange to hexane is required, add approximately 30mL (mix well) and return to concentrator set at 35°C. See Attachment 2.

10.1.17 Total concentration time should be approximately 20 minutes for methylene chloride. If a sample takes a longer than usual amount of time to concentrate or if the extract becomes viscous the final volume should be adjusted to a larger volume and documented.

10.1.18 The extract is now ready for the appropriate clean up and or analysis.

10.2. Calibration

10.2.1 The balance calibration is checked daily prior to use. Refer to TestAmerica Edison SOPs ED-GEN-010, *Calibration of Analytical Balances*, current revision and ED-GEN-006, *Standard Operating Procedure for Preventive Maintenance and Calibration Procedures for All Analytical Instruments and Ancillary Equipment*, current revision.

11.0 Calculations / Data Reduction

N/A

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0 Pollution Control

13.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs No. ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- *Mixed Solvent Waste*: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- *Auto sampler vials and expired standards*: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, “Method SW3546, Microwave Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Laboratory Manual, Physical/Chemical Methods, February 2007.

15.2 United States Environmental Protection Agency, “Method SW8015B, Non-Halogenated Organics, Test Methods for Evaluating Solid Wastes, SW846 Third Edition,Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.3 United States Environmental Protection Agency, Method SW8015C, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, February 2007.

15.4 United States Environmental Protection Agency, Method SW8015D, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 4, June 2003.

15.5 NJDEP Method No. OQA-QAM-025, “Quantitation of Semivolatile Petroleum Products in Water, Soil, Sediment and Sludge” (current revision)

15.6 United States Environmental Protection Agency, “Method 8081A, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.7 United States Environmental Protection Agency, “Method 8082, Polychlorinated Biphenyls PCBs) by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.8 United States Environmental Protection Agency, “Method 8081B, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, February 2007.

15.9 United States Environmental Protection Agency, “Method 8082A, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 1, February 2007.

15.10 United States Environmental Protection Agency, “Method SW8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.11 United States Environmental Protection Agency, “Method SW8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 4, February 2007.

15.12 TestAmerica Edison SOP No. ED-GCS-008, *Preparation and Analysis of Diesel Range Organics (DRO C10-C28) in Soil and Water Samples by SW846 Methods 3510C (Separatory Funnel), 3541 (Automated Soxhlet Extraction) and 8015B(GC/FID),* current revision.

15.13 TestAmerica Edison SOP No. ED-GCS-009, *Preparation and Analysis of Diesel Range Organics (DRO C10-C44) in Soil and Water Samples by SW846 Methods 3510C (Sep Funnel), 3541 (Automated Soxhlet) and 8015B (GC/FID),* current revision.

15.14 TestAmerica Edison SOP No. ED-GCS-011,*NJDEP OQA-QAM-025 Quantitation of Semivolatile Petroleum Products in Water Soil Sediment and Sludge,* current revision.

15.15 TestAmerica Edison SOP No. ED-GCS-003, *Analysis of Organochlorine Pesticides by Gas Chromatograph, SW846 Method 8081A*, current revision.

15.16 TestAmerica Edison SOP No. ED-GCS-004, *Analysis of Polychlorinated Biphenyls by Gas Chromatograph, SW846 Method 8082*, current revision

15.17 TestAmerica Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by Gas Chromatograph, SW846 Method 8081B*, current revision.

15.18 TestAmerica Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by Gas Chromatograph, SW846 Method 8082A*, current revision

15.19 TestAmerica Edison SOP No. ED-MSS-010, *Determination of PCBs in Water and Soil/Sediment by GC/MS, EPA Method 680,* current revision.

15.20 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.21 TestAmerica Environmental Health and Safety Manual, CW-E-M-001.

15.22 TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing& Approval*, current revision.

15.23 TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.24 TestAmerica Edison SOP No. ED-GEN-013, *Glassware Cleaning,* current revision

15.25 TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision

15.26 TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures,* current revision.

15.27 TestAmerica Edison SOP No. ED-GEN-006, *Standard Operating Procedure for Preventive Maintenance and Calibration Procedures for All Analytical Instruments and Ancillary Equipment,* current revision.

15.28 TestAmerica Edison SOP No. ED-GEN-010, *Calibration of Analytical Balances,* current revision.

15.29 TestAmerica Edison SOP No. ED-GEN-007, *Subsampling*, current revision.

15.30 Operation Manuals for the Zymark Turbo Vap II Concentrator.

15.31 Operating manual for the MARS 5 Microwave unit.

16.0 Method Modifications:

N/A

17.0 Attachments

Attachment 1: Spiking information
Attachment 2: Specific Extraction Conditions

18.0 Revision History

Rev 11, March 12, 2018

- Updated Section 10.1 to revise procedures for the microwave extraction of solids for subsequent analysis by EPA methods 8081, 8082 and 8270.
- Updated to remove all references to addition of sodium sulfate to samples prior to microwave extraction.
- Fixed section numbering and formatting errors.

Rev 10, May 16, 2017

- Updated throughout to add procedures for microwave extractions of solids for subsequent analysis by EPA Method 680 (PCBs by GC/MS).

Rev 9, Dec 15, 2016

- Updated throughout to remove procedures for microwave extraction of solids for NJ EPH method. Those procedures are fully covered in TestAmerica Edison SOP ED-GCS-012, Preparation and Analysis of Extractable Petroleum Hydrocarbons (EPH) in Solid and Water Samples using NJDEP EPH Method 10/08, August 2010 (Rev. 3): Analysis of Extractable Petroleum Hydrocarbon Compounds (EPH) in Aqueous and Soil/Sediment/Sludge Matrices (current revision)
- Added following text to Section 1.2: The microwave prep for NJDEP Extractable Petroleum Hydrocarbons (EPH) (10/08), current revision, is addressed separately in TestAmerica Edison SOP ED-GCS-012 (current revision).
- All references to NJDEP EPH methodology removed.

Rev 8, Jan 22, 2015

- Updated to include procedures for microwave extraction of solids for analysis my SW846 Methods 8270C and 8270D (Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS). Specifically
    - Added 8270 method references and SOP numbers to Sections 1.1 and 15.0
    - Added a table to Section 7.2.1 detailing the BNA standard mixes.
    - Added new Section 7.2.4 detailing the preparation of BNA spiking standards

- Added Section 7.2.4.1 detailing the preparation of BNA surrogate spiking standards.
- Added details of 8270 spiking and extraction to Section 10.0 as appropriate.
- Added BNA Spiking details to Attachments 1 and 2. COAs for BNA spiking not included in SOP but are on-file in the department.

Rev 7, June 14, 2013

- Section 1.1: updated the DRO method references to include SW8015C and SW8015D. Also update the SOP references for the DRO analytical methods.
- Section 7.2.1: updated stock standards table to include SW8015C and 8015D. Revised source for o-Terphenyl stock. Removed TPH Mix 1 from list. Removed 8015x DRO from the list of methods for which TPH Mix 3 is used. Added 8015x DRO to the list of methods for which Diesel Fuel #2 is used.
- Section 7.2.2.1: Revised DRO surrogate spike source and prep instructions. Revised DRO and QAM-025 spiking standard source and prep instructions.
- Section 15.0: References: added references for methods SW8015C and 8015D.
- Attachment 1: updated spiking information for DRO.

Rev 6, October 10, 2012

- Throughout document:: Revised LQM reference sections to reflect the most current revision
- Throughout document: updated as detailed below to include prep instructions for solid samples undergoing analysis by SW846 8081A/B and 8082/A.
- Section 1.1: added references to the 8081/8082 SOPs: TestAmerica Edison SOPs No.ED-GCS-003, ED-GCS-004, ED-GCS-016 and ED-GCS-017.
- Section 6.2: added weigh boats and wooden tongue depressors.
- Section 7.2.1: added acetone as a diluent and added tables detailing the Pestcide and PCB standards.
- Section 7.2.2: added “GCFID (DRO/EPH/QAM)” to section header.
- Section 7.2.3: added instructions for prep of Pest/PCB spiking standards.
- Section10.1.4: Added Pest/PCB spiking instructions.
- Section 10.1.5: added instructions for addition of extraction solvent (Methylene Chloride:Acetone for Pest/PCB).
- Section 10.1.8: updated table to include Microwave Parameters for Pest/PCB prep.
- Section 10.1.12: added a methylene chloride rinse.

- Section 15: added method and SOP references for SW846 8081A/8081B and SW846 8082/8082A.
- Attachments: updated to include Pest/PCB information.

Rev 5, August 26, 2011

- Section .6.2: added vendor and part number details for silica gel columns (Phenomenex Part No:8L-S012-JCH)
- Section 10.1.2: Added details for the pre-washing of sodium sulfate funnels with three successive 30 ml volumes of acetone. Added details for the rinsing of the 75 ml vessel with three successive volumes of methylene chloride.

Rev 4, April 21, 2010

- Section 7.2.1: Corrected the footnotes in the tables 'Stock Standards – Aliphatics (Method NJDEP EPH 10/08)' and 'Stock Standards-Aromatics (Method NDJEP EPH 10.08)'.
- Attachment 3: Added this attachment which consists of select certificates of analysis for Method NJDEP EPH 10/08.
- Section 7.2.2.1: Table 'Working Spiking Standards Preparation'" replaced asterisks (*) with dashes (-) in order to clarify that these are simply bullets rather than referencing footnotes.

Rev 3, April 19, 2010

- Section 7.2.2.1: added procedure for preparation of a 500 ppm Aliphatic Neat Spike Mix.
- Section 7.2.2: Spiking Standards Prep: revised the procedure for prep of the EPH Aliphatic Spike in the 'Working Spiking Standards Preparation' table as follows:
    - Replaced 'MA EPH-n-Hydrocarbons (Absolute/91488) with the 'Florida TRPH Standard (Restek/31266)'
    - Included used of the intermediate 500 Aliphatic Neat Spike Mix.

Rev 2, February 9, 2009

- Section 3.0, Definitions – updated to reflect current location of definitions (Appendix 2 of Lab Quality Manual
- Section 5.2, Primary Materials Used – added 'hexane' to table (used in the NJDEP EPH method).
- Section 7.2.1: added stock standards associated with NJDEP EPH 10/08 method.
- Section 7.2.2: Spiking Standards Prep: updated 'Working Spike Standards' table to include revised NJDEP EPH 10/09 spiking standards; revised other as required.
- Section 9.1, Sample QC: updated with revise NJDEP EPH 10/09 QC requirements.
- Section 10.1, Sample Preparation: revised initial sample weight to 15 grams.
- Section 10.1.8: added Microwave Operating Parameters for the NJDEP EPH 10/09 method.
- Section 10.2.1: changed the specifications of the silica column.

- Section 10.2.5: changed the volume of the hexane rinsate.
- Section 10.2.7: changed the volume of the methylene chloride rinsate.
- Section 10.2.11: Added text describing procedures for NJDEP EPH 10/09 Demonstration of Fractionation Capability.
- Section 14.1 and Section 15.0 : Removed reference to TestAmerica Edison SOP No. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* (SOP is now retired).
- Section 15.0: updated references to NJDEP EPH method as well as TestAmerica Edison SOP for NJDEP EPH analysis.
- Section 17.0, Attachments: corrected descriptions of attachments.

Rev 1, June 29, 2009

- Section 1.0, Scope and Application – corrected typo in the temperature range 100 – 115°C
- Section 5.2, Primary Materials Used – removed ‘sodium hydroxide’ and ‘sulfuric acid’ from table as they are not used in this method.
- Section 6.1, Instrumentation – added Visiprep DL column holder to the instrumentation list
- Section 6.2, Supplies – added 5 g silica gel column (20 ml capacity)
- Sections 7.2.1, 7.2.2, Attachment 1 and Attachment 2: corrected a typo in the abbreviation for Methylene Chloride ($MeCl_2$).
- Section 10.1.1.2 - sentence correction to show actual process and typo error correction. Phrase ‘may also’ changed to ‘shall’.
- Section 10.1.12 – replaced the word ‘salt’ with ‘Sodium Sulfate’ to show actual reagent being used.
- Section 10.1.17 – typo error correction to ’20 minutes’
- Section 10.2.5 – Added the following text to the end of the section: “The amount on hexane will need to be adjusted with every lot of silica columns. The Hexane amount should never exceed 20 mls.”
- Corrected carbon ranges for the reference DRO SOPs throughout the document.

Rev 0, March 27, 2009: New

Attachment 1

Spiking Information – Microwave Extraction

Method	Surrogate Amount (all samples and QC)	Spike Amount (LCS/MS/ MSD)	Final volume	Initial Solvent	Final Solvent
DRO (8015)	1 mL	1 mL	1 mL	$MeCl_2$	$MeCl_2$
QAM-025	1 mL	1 mL	1 mL	$MeCl_2$	$MeCl_2$
Pest/PCB (8081/8082)	0.05 mL	0.1/0.05 mL	10 mL	MeCl2/Acetone (50:50)	Hexane Exchange from MeCl2/Acetone
BNA (8270)	500uL	500uL	1 mL	MeCl2/Acetone (50:50)	$MeCl_2$
PCB Homologues (EPA 680)	1.0	1.0	1 mL	Hexane	Hexane

Attachment 2

Specific Extraction Conditions

Method	Initial extraction pH	Secondary extraction pH	Final Solvent	Volume of extract required for cleanup(mL)	Final extract volume for analysis (mL)
DRO (8015)	as received	NA	$MeCL_2$	NA	1.0
QAM-025	as received	NA	$MeCL_2$	NA	1.0
Pest/PCB (8081/8082)	as received	NA	Hexane	3	10.0
BNA (8270)	as received	NA	$MeCL_2$	NA	1.0
PCBs (EPA 680)	as received	NA	Hexane	NA	1.0

Title: Closed System Purge and Trap and Extraction for Volatile Organics in Soil , SW846 Method 5035A

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method describes a closed system purge and trap procedure for the analysis of volatile organic compounds in soils, sediments and solid wastes.

1.1.2 This method is designed for use on samples containing low levels of volatile compounds (0.5 to 200ug/kg), and requires the use of hermetically sealed sample vials, which limits a samples exposure to the atmosphere, thereby minimizing the loss of volatile compounds.

1.1.3 Method 5035A can be used for most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Water-soluble compounds can be included in this technique, but will have higher quantitation limits due to poor purging efficiency.

1.1.4 Method 5035A also includes sample collection and preparation procedures for medium and high concentration soil samples (i.e., >200 ug/kg). Medium and high level soil samples are prepared either by field preservation of a soil sample in methanol or by creating a methanol extract from a soil sample that was collected as bulk soil.

1.1.5 The preparation steps described for medium and high level samples will be followed by the aqueous purge and trap procedure detailed in TestAmerica Edison SOP No. ED-MSV-001(*Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*).

1.1.6 The preparation and sample introduction steps described for low-level soils will be followed by analysis using TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) or ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*) as applicable.

1.1.7 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 **Low Level Soils:** Volatile organic compounds are determined by collecting a 5g-soil sample with a specially designed sampling device (ex., En Core® Sampler) in which the sample is stored without headspace. Upon receipt and within 48 hours of sampling, the soil is placed in a pre-weighed vial containing 5 mL of reagent water and a stir bar. The vial containing the soil is then frozen extending the hold time to 14 days. The same samples may be received from the field in a 40 mL vial with 5 mL water (ex., Terra Core® Sampler). Immediately prior to analysis, the soil is thawed, reagent water, surrogate spiking solution and/or internal standard

spiking solution are added to the vial by the automatic sampler, without opening the vial to the atmosphere. The sample vial is heated and purged with helium while being magnetically stirred. The volatile components travel through a transfer line to a sorbent trap. When purging is complete, the trap is heated and backflushed with helium to desorb the sample components on to a GC column for separation and analysis by the appropriate determinative method.

2.2 **Medium Level Soils:** Medium level soils are soils that were originally intended to be analyzed by a low level soil method, but show contaminants >200ug/kg when screened. A methanol extract is prepared by extracting 5g of soil with 10mL of methanol. The volatiles are effectively transferred from the soil to the methanol. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

2.2.1 Samples may also arrive from the field as 5 mL or 10 mL methanol preserved samples. These are considered to be medium level prepared samples.

2.3 **High Level Soils:** The high level soil method acts as a combined preservation and extraction technique. 25mL of methanol /surrogate solution is placed in each VOA vial and weighed prior to field sampling. Approximately 10g of soil is added to each vial in the field. Upon returning to the lab, the vial is weighed again, and the difference between the two weights is recorded as the initial weight of sample. As with the medium level method, the volatiles are effectively transferred to the methanol. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

2.4 **Waste Dilution:** Organic waste samples are prepared by extracting 1g of sample with 10mL of methanol / surrogate solution. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method and the determinative methods that follow are susceptible to contamination from a number of sources. Potential sources of contamination include organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatile laboratory is located in a separate building, away from the organic extraction area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatile laboratory.

4.3 The nitrogen used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 A trip blank prepared from organic-free reagent water is carried through the sampling, storage, and analysis of each group of samples to check for such contamination.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Purge vessels are removed from the autosampler units after each use, rinsed, baked, returned to the units and pre-purged before the next use.

4.7 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.8 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

4.9 Potential cross-contamination of samples stored in lab refrigerators is monitored by preparation, analysis and evaluation of storage blanks. Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After one week, the storage blanks are removed and analyzed. Additional details can be found in TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

5.1.1 Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required. A purge and trap unit consists of three parts: the sample purge unit, the trap, and the concentrator. Unit configurations currently in use are:

- OI Analytical 4551 Automatic Sampler/4560 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4560 concentrator;
- EST Centurion Autosampler/ EST Encon concentrator;
- Archon Autosampler/EST Encon concentrator.

6.1.2 A VOCARB 3000 trap from Supelco is used in the Encon concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001

6.1.3 An OI analytical purge trap #10 is used for the OI 4560 concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed to contain the following absorbents: Tenax/silica gel/carbon molecular sieve.

6.1.4 Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 Both the Encon and OI concentrators are capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.6 Gas chromatograph: HP 5890/Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: reference the applicable determinative method for column specifics.

6.1.8 Detector: Flame Ionization Detector or Mass Spectrometer (HP5971/5972/Agilent 5973). Reference the applicable determinative method for detector specifics.

6.1.9 Data system: HP Chemstation II for data acquisition and HP UNIX based TARGET software for data processing.

6.2 Equipment and Supplies

6.2.1 Freezer: Capable of holding a temperature of -7℃ to -20℃.

6.2.2 Refrigerator: Capable of holding a temperature of 4℃ ±2℃

6.2.3 Top loading analytical balance.

6.2.4 Portable field balance. Capable of weighing to 0.01g.

6.2.5 Microsyringes: 10 uL to 1000 uL.

6.2.6 Syringes: 5mL, 10mL, and 30mL gas-tight.

6.2.7 Volumetric flasks: Class “A” glassware, 10mL , 50mL , and 100 mL.

6.2.8 VOA vials: 40 mL glass with PTFE –faced septum.

6.2.9 En Core® Sampler. Designed to take a 5g-soil sample. Sealed to prevent loss of volatiles.

6.2.10 Vials: 2mL amber glass with screw cap with Teflon-faced septa.

6.2.11 Spatula: Narrow, stainless steel.

6.2.12 Stir bars: PTFE coated, small enough to spin freely inside a VOA vial.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Organic free reagent water. Distilled water purchased from Poland Spring.

7.1.2 Methanol. Purge and trap grade, purchased from JT Baker. (Cat # 9077-02)

7.1.2.1 Each lot of methanol and hydrochloric acid is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2 Standards

7.2.1 Calibration standard preparation and use is described in detail in each of the determinative methods.

7.2.2 Method 8260 Methanol/Surrogate solution (2.5 ppm) is prepared using purge and trap grade methanol and neat a,a,a-Triflurotoluene solution. Initially a primary solution is prepared at 2500 ppm:

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume of MeOH
1°Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4	Sigma Aldrich	 B67201 151998 396540	 1585 ul 2678 ul 1932 ul	Neat	2500ppm	1000 ml

The 2500 ppm solution is used to prepare a 2.5 ppm Method 8260 Methanol/Surrogate working solution by diluting 1.0 ml of 2500 ppm stock to 1000ml with purge and trap grade methanol.

7.2.3 Method 8015 (GRO) Methanol/Surrogate solution (1.25 ppm) is prepared by diluting 0.5ml of the 2500 ppm stock (see Section 7.1.4) to 1000 ml with purge and trap grade methanol.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Low Level Soils

Sample containers used to collect low-level soils are 40 mL VOA vials with PTFE-faced septa (closed system purge and trap), En Core® Samplers, Terra Core® kits or bulk soil.

8.1.1 For the 40 mL VOA vial (closed system purge and trap) collection procedure, add 1 clean magnetic stir bar and 5 mL of reagent water to each vial.

8.1.1.1. Cap the vial and weigh to the nearest 0.01g. Record this as the tare weight.

8.1.1.2. Collect approximately 5g of soil using an appropriate sampling device and add 5g to each of 3 vials for low level analysis.

8.1.1.3. Collect a fourth aliquot for field methanol preservation into a pre-weighed vial containing 10 mL of methanol.

8.1.1.4. Closed system purge and trap collection vials are re-weighed to determine actual sample weight. Record the sample weight on the sample vial.

8.1.1.5. Within 48 hours of sampling the collection vials are placed into a freezer (on their side to prevent breakage) at -7°C to -20°C. Freezing in this manner extends the holding time to 14 days from sampling.

8.1.2 For En Core® Sampler collection, collect up to 3 En Core® Sampler for low level preservation via freezing and a fourth for medium level preservation with methanol (Section 8.2 details the medium level preservation procedures for En Core® samplers). The low level preservation procedures are:

8.1.2.1. The En Core® device is received at the lab sealed without headspace with a cap and viton o-rings.

8.1.2.2. For each En Core® requiring low level preservation, add 5ml of reagent water and a magnetic stir bar to a 40 ml vial. Cap the vial.

8.1.2.3. Weigh each vial to the nearest 0.01g. Record this as the tare weight.

8.1.2.4. Transfer the contents of each En Core® to separate preweighed vials within 48 hours of sampling.

8.1.2.5. Reweigh the vial. Record the weight on each sample vial.

8.1.2.6. The vials are placed into a freezer (on their side to prevent breakage) at -7°C to -20°C. Freezing in this manner extends the holding time to 14 days from sampling.

8.1.2.7. A bulk soil sample must also be collected for the purposes of percent moisture analysis for dry weight determination.

8.1.2.8. Vials are removed from the freezer the day of analysis for thawing.

8.1.2.9. Stir bars may be reused after the vial is analyzed. Retrieve the stir bar from each vial using the magnetic stir bar retriever, rinse the stir bar with methanol and bake at 105°C for 2 hours.

8.1.3 Terra Core® Kits. This option requires samples to be field preserved:

8.1.3.1. The sample kit typically includes two pre-weighed 40 mL vials containing 5 mL of water with a small magnet stir bar and one or two pre-weighed 40 mL vials containing pre-measured and recorded amount of methanol.

8.1.3.2. Approximately 5g of sample is placed into each vial by the field sampler.

8.1.3.3. The vials containing the samples are re-weighed in the lab and the net sample weight is recorded for each vial. The vials containing the sample and water are frozen up until the time of analysis.

8.1.3.4. The methanol preserved samples are considered to be medium level prepared samples and do not require freezing but they must be refrigerated at 4℃ until time of analysis.

8.1.3.5. A bulk soil sample must also be collected for the purposes of percent moisture analysis for dry weight determination.

8.1.3.6. Stir bars may be reused after the vial is analyzed. Retrieve the stir bar from each vial using the magnetic stir bar retriever, rinse the stir bar with methanol and bake at 105°C for 2 hours.

8.1.4 Bulk soil. Typically consists of soil collected in 8-oz or 16-oz glass jars. At client request bulk soil samples can be sub-sampled, preserved and analyzed for volatile organics. Soils collected for low level VOA analysis as bulk soil in jars will be preserved as described above in Section 8.1.1. Documentation of preservation is recorded in the Soil Preservation Logbook.

8.2 Medium Level Soils

8.2.1 Medium level refers to any soil sample extracted in methanol not preserved in the field. The fourth Encore sample collected (as described in Section 8.1.2 above) will be prepared as a medium level soil in case the analysis of a low level preserved aliquot exhibits concentrations of target compounds exceeding 200 ug/kg. All medium level extracts must be refrigerated at 4℃ until time of analysis.

8.2.1.1. Prepare the medium level soil by adding 5 g of soil or Encore to 10 mL of Methanol / Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5. depending upon the method to be analyzed). All medium level extracts shall be prepared within 48 hours of collection. The weight of soil used to prepare the extract is recorded on the sample vial.

8.2.1.2. Since methanol extract results tend to increase with time as the sample contact time increases, a minimum contact time of one day is allowed ***or*** the soil is sonicated for 20 minutes.

8.2.1.3. Any soil requiring medium level preservation but collected out of compliance with method 5035A (bulk soil without preservative or not in an Encore device) will be prepared in the same manner as in section 8.2.1.1. Documentation will be noted in the soil preservation log.

8.3 High Level Soils

8.3.1 High level soils are field collected using laboratory prepared 40 ml VOA vials (with PTFE-faced septa) containing 25 mL of the Methanol/Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5. depending upon the method to be analyzed).

8.3.2 The vials containing the Methanol/Surrogate solution are weighed using a barcode reader and an analytical balance. The bottle ID and weight (Weight 1) are recorded in the methanol-tracking program.

8.3.3 The containers are issued to the field as required.

8.3.4 Field samplers are directed to weigh 10 g of sample using a field balance. They must carefully add the 10 g sample to the sample vial containing the Methanol/Surrogate solution, taking care not to spill any of the solution from the vial.

8.3.5 Field samplers are directed to wipe the neck of the vial carefully to remove any soil around the threads. This helps to ensure a good seal once the vial is capped. Field samplers are further directed to cap the vial, shake gently, dispersing the soil into the methanol. An additional aliquot of soil without any methanol or preservative must be collected for use in determining percent moisture (i.e., for dry weight determination).

8.3.6 Upon the return to the laboratory, the sample vials (now containing both the Methanol/Surrogate solution and approximately 10g of soil) are reweighed using the barcode reader and analytical balance. Record this weight (Weight 2) in the methanol tracking system. Calculate the initial weight of soil by subtracting Weight 1 from Weight 2.

8.3.7 All samples for volatile analysis are protected from light and stored at 4°C±2°C in an area free of organic solvents. No standards or solvents are stored in the sample refrigerators or in the surrounding area.

8.3.8 All samples preserved in this manner must be analyzed within 14 days of collection.

9.0 Quality Control

9.1 Specific quality control procedures are outlined in each of the determinative methods that follow this purge and trap technique of sample introduction. Standard quality assurance practices are used with all methods.

9.2 Reagent water blanks are analyzed to ensure that reagents and/or sample dilutions are free of interferences.

9.3 Method blanks are prepared and analyzed in the same manner as samples, and are carried through the entire analytical procedure to show that each system is in control.

9.4 Potential cross-contamination of samples stored in lab refrigerators is monitored by preparation, analysis and evaluation of storage blanks. Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After one week, the storage blanks are removed and analyzed. Additional details can be found in TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

9.5 An initial demonstration of accuracy and precision is performed for each determinative method. This demonstration is done for each sample introduction technique.

10.0 Procedure

10.1 This section provides guidance for the analysis of low level soils using a closed system purge and trap procedure. Reference the SOPs for the determinative methods for additional details as applicable: TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) or ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*).

10.2 Instrument operating parameters are set at the beginning of a method of analysis and remain constant throughout the entire analytical procedure. Again, reference the SOP for the applicable determinative method for specifics. Sample operating conditions are listed here:

Example Instrument Operating Parameters

Purge and trap unit	
Purge Time	11 minutes
Dry Purge	1 Minutes
Purge Gas	Helium
Purge Flow	40-45 ml/min
Purge Temp	Water - Ambient; Solids - 40^{0}C
Trapping Temp	Ambient, <30^{0}C
Desorb Time	1-2 Minutes
Desorb Temp	Vocarb - 260^{0}C
Gas chromatograph	
Injector	180^{0}C
Carrier Gas	Helium
Carrier Flow	6 ml/min
Oven Program	35 - 250^{0}C
Run Time	30 Minutes
Mass Spectrometer	
Electron Energy	70 volts (nominal)
Mass range	35-260 AMU
Scan time	0.9 sec./scan
Source Temp	200^{0}C
Separator Temp	180^{0}C

10.3 All samples are screened prior to analysis, using the procedure outlined in TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID SW846 Method 5021(modified),* current revision. In addition to reducing excessive instrument contamination and/or extensive instrument clean up, screen data provides the analyst with an approximate dilution factor and is used to determine which sample preparation technique should be used for a particular sample. If the estimated concentration from the screening procedure shows concentrations below 200 ug/kg, follow the low concentration closed system purge and trap method. If the screen result shows concentrations >200 ug/kg, follow the medium level procedure.

10.4 Low Level Soil Procedure

10.4.1 Low level soils are field collected as described in Section 8.1 using 40 mL VOA vials with PTFE-faced septa (closed system purge and trap), En Core® Samplers, Terra Core® kits or bulk soil.

10.4.2 Set up the purge and trap autosampler (Archon 5100A or EST Centurion, see Section 6.1) for the closed system method for low-level soils. The purge and trap autosampler will add 5 mL reagent water, and 1uL of the internal standard and/or surrogate spiking solution to the sample vials. The heating block will heat each sample to 40° C and hold for 0.5 minutes before purging begins.

10.4.3 Tuning, calibration, and method blank analysis:

- Tuning and calibration is accomplished as detailed in the SOP for the applicable determinative method.

- The volume of water used when purging a calibration standard must be the same as the volume of water purged during a sample analysis. Since the low-level soils have 5 mL water added at vial preparation plus the 5 mL of reagent water added by the purge and trap autosampler prior to purging, a total volume of 10 mL is required for calibration standards.

- Internal standard (if required by the determinative method) is added to each calibration standard by the purge and trap autosampler, in the same manner it will be added to the samples.

- After successful calibration, analyze a method blank. The method blank must meet all criteria in the determinative method. A successful method blank shows that the system is free of interferences and sample analysis may begin.

10.5 Medium Level Soil Procedure

10.5.1 Medium level soils are typically field collected using laboratory prepared 40 ml VOA vials (with PTFE-faced septa) containing 25 mL of the Methanol/Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed).

10.5.2 When preparing a medium level soil from a bulk soil sample, do not decant any supernatant liquids. Quickly mix the contents of the sample container with a narrow spatula. Weigh out 5g of soil into a 40ml vial, add 10ml of Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) and cap the container and proceed with Section 10.5.6.

10.5.3 For samples collected in the Encore sampling device, break open one end and push the aliquot of soil out into a vial. Add 10ml of the Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) and cap the vial and proceed with Section 10.5.6.

10.5.4 For Method 8260 samples collected as Terra Cores® in 5 mL or 10 mL methanol add 5 uL and 10 uL of the 2500 ppm 8260 Methanol/Surrogate mix (see Section 7.1.4) respectively to extract and proceed with Section 10.5.6.

10.5.5 For Method 8015 (GRO) samples collected as Terra Cores® in 5 mL or 10 mL methanol add 2.5 uL and 5 uL of the Methanol/Surrogate Mix surrogate solution (see Section 7.1.5) respectively and proceed with Section 10.5.6.

10.5.6 Cap and shake the extract for 2 minutes. Allow soil to settle.

10.5.7 Transfer 2 mL of the extract to a 2 mL amber glass vial for storage with minimal headspace.

10.5.8 Refrigerate at 4°C until the time of analysis.

10.5.9 Analyze a portion of this extract using Method 5030 purge and trap for aqueous samples (TestAmerica Edison SOP No. ED-MSV-001, *Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*). The appropriate dilution factor is calculated from the low-level analysis.

10.6 High Level Soil Procedure

10.6.1 High level soils have been preserved in the field by sampling either 10 g of soil into vials containing 25 mL Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) or 5 g of soil into vials containing 10 mL Methanol/Surrogate mix solution (again, either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed).

10.6.2 Screen high level soils by Method 5021 to determine the proper dilution factor prior to analysis (see TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics,Static Headspace with GC FID SW846 Method 5021(modified),* current revision.)

10.6.3 Transfer 2 ml of the extract to a 2 mL amber glass vial for storage with minimal headspace.

10.6.4 Analyze a portion of this extract using Method 5030 purge and trap for aqueous samples.

10.7 Sample Analysis

10.7.1 Remove sample vials from storage and allow thawing to ambient temperature before analysis. Shake the vial gently to ensure that the contents move freely within the vial. Place the sample vials in the autosampler tray.

10.7.2 Schedule the autosamplers to run the samples using the low level soil method. 5 mL of reagent water plus 1 uL of internal standard/ surrogate spiking solution is added to the sample vial directly through the septum.

10.7.3 Prior to purging, the sample is heated to 40°C for 0.5 minutes.

10.7.4 Purge the sample with helium for 11 minutes while agitating with magnetic stirring.

10.7.5 The purgeable compounds will flow out of the vial through a glass lined transfer line to a sorbent trap.

10.7.6 Desorb the contents of the trap on to the GC column by rapidly heating the trap to 260 °C and backflushing it with helium.

10.7.7 Begin the GC temperature program and data acquisition.

10.7.8 Re-condition the trap after desorb by baking at 260° C for 8-12 minutes.

10.7.9 Perform quantitative and qualitative analysis using TARGET software. Evaluate data according to each determinative method.

10.7.10 If a re-analysis is required, perform the re-analysis using the second frozen sample vial.

10.7.11 If a dilution is required, analyze the methanol preserved aliquot by Method 5030.

10.7.12 Technical acceptance criteria for sample analysis. (Note: these are general guidelines; please see the SOP for the determinative method for more specific acceptance criteria)

- The samples must be analyzed on a GC or GC/MS system meeting the initial calibration, continuing calibration and blank technical acceptance criteria.
- The sample must be analyzed within the required holding time.
- The sample must have an associated method blank meeting the blank technical acceptance criteria.
- The percent recovery of each of the system monitoring compounds in the sample must be within the acceptance windows.
- After analyzing a sample that exceeds the initial calibration range the analyst must either analyze an instrument blank (using the same purge inlet if using an auto sampler) which must meet technical acceptance criteria for blank analysis or monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample that exceeded the calibration range.

10.7.13 Corrective Action for Sample Analysis (Note: these are general guidelines; please see the SOP for the determinative method for more specific corrective actions):

- Samples must meet technical acceptance criteria before reporting data.

- Corrective action for failure to meet instrument performance checks, initial, continuing calibration and method blanks must be completed prior to sample analysis.

- Corrective action for system monitoring compounds and internal standard compounds that fail to meet acceptance criteria must be completed prior to sample analysis.

- If any of the system monitoring compounds and internal standard compounds fail to meet acceptance criteria, check all calculations, instrument logs, the system monitoring compound and internal standard compound spiking solutions and the instrument operation. If the calculations were incorrect, correct calculations and verify that the system monitoring compound recoveries and internal standard compound responses meet acceptance criteria.

- Check the preparation of the internal standards and system monitoring compounds for concentration and expiration.

- Verify that the instrument is operating correctly.

- Data that fails to meet minimum acceptance criteria will be annotated (flagged) with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Reports will be initiated in order to provide for investigation and follow-up.

11.0 Calculations / Data Reduction

11.1 Methanol volume correction for soil moisture content (medium/high level methods):

$$Vt\ (\mu L\ solvent/water) = [\ ml\ of\ solvent + \frac{(\%moisture \times g\ of\ sample)}{100}] \times 1000\ uL/mL$$

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training* (latest revision) for the laboratory's training program.

13.0 Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out.

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system**.**

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These

boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

Teris Profile Number (incineration): 50016710
Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

- Returned Methanol Preservative - Methanol preserved vials are collected in satellite accumulation and then transferred to 55 gallon open top steel waste drums in the waste room. These waste drums are then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, *Method 5035A: Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 0, December 1996.

15.2 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3 TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics,Static Headspace with GC FID SW846 Method 5021(modified),* current revision.

15.4 TestAmerica Edison SOP No. ED-MSV-001, *Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*.

15.5 TestAmerica Edison SOP Nos. ED-GCV-006, *Gasoline Range Organics using GC FID Method SW846 8015, current revision.*

15.6 TestAmerica Edison SOP No ED-MSV-005, *SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision.*

15.7 United States Environmental Protection Agency, *Method 8000C: Determinative Chromatographic Determinations*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, March 2003

15.8 TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

15.9 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001)

15.10 TestAmerica SOP No. ED-GEN-022, *Training* ,latest revision.

15.11 TestAmerica Edison SOPs Nos. ED-SPM-007, *Disposal of Samples and Associated Laboratory Waste, current revision.*

15.12 TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures,* current revision.

16.0 Method Modifications:

16.1 N/A

17.0 Attachments

17.1 N/A

18.0 Revision History

- Revision 10, effective date: 4/11/2019
  - Section 8.2.1: Added temperature preservation requirement for Medium level extracts.

- Revision 9, effective date: 11/27/2012
  - Updated referenced Lab Quality Manual section and appendix numbers as appropriate

- Revision 8, effective date: 12/6/2010
  - Section 3: revised to reference new location for definitions.
  - Sections 8.2.1.4 and 8.3.9 referencing methanol solvent adjustment for solid samples with significant moisture content (>10%) was removed.

- Revision 7, effective date: 11/4/2008:
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Updated the SOP to comply with SW846 Method 5035A (was 5035). Retitled SOP accordingly.
  - Section 1.1.5: Added reference to TestAmerica Edison SOP No. ED-MSV-001(*Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*).
  - Section 1.1.6: Added references to TestAmerica Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current*

revision) and ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*)

- Section 1.1.7: Added reference to Quality Assurance Manual for method modifications.
- Section 3: revised to reference new location for definitions.
- Section 4: revised to include a reference to storage blanks and the applicable SOP: TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination*, current revision.
- Section 5 (Safety): Revised to include most up to date corporate health and safety references and information.
- Section 6: Updated with current instrumentation and configurations.
- Section 7.0: added details of the solvent testing and approval program.
- Section 7.2: Updated standards sources and catalog numbers.
- Section 8: Updated with additional details for each type of preservation/analysis (low level, medium level, high level). Added used of Terra Core® Sampling Kits (Section 8.1.3).
- Section 8: Updated to include requirement to adjust the methanol volume (medium/high level) for soil moisture content.
- Section 9.0: Added details of storage blanks and reference to TestAmerica Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision..
- Section 10: Revised and expanded to include instrument operating conditions and specific sample prep details for low level, medium and high level preps.
- Section 11: Deleted reference to SOP for Organic Calculations. Added statement that calculations can now be found in the applicable determinative methods.
- Section12.0: added reference to Training SOP.
- Section 13.0: Revised to include most up to date company policy on Pollution Control as well as to include corporate health and safety references and information.
- Section 14.0: Revised to include most up to date company policy on Waste Management as well as to include corporate health and safety references and information.
- References: Expanded to include more specific SOP references.
- Revision history: updated.

Title: The Analysis of Waters for Hexavalent Chromium by EPA SW846 Method 7196A

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1. SW846 Method 7196A may be used to determine the hexavalent chromium content of groundwater samples or digestates of solid samples. This SOP is applicable for the determination of hexavalent chromium in groundwater samples. Groundwater samples associated with regulated drinking water samples (i.e. potable water wells) are not applicable to this Method.

1.2. The range of analysis by this method is 10-200 ug/l hexavalent chromium.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Hexavalent chromium is determined spectrophotometrically at 540nm by reaction with diphenylcarbazide in acid solution. For each batch of samples a method blank, a laboratory control sample, matrix duplicate, and a post verification spike are analyzed and compared to method limits and laboratory control limits.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Molybdenum and Mercury react to form color with diphenylcarbazide, however the intensity of color is much lower than those for chromium at the specified pH. Concentrations up to 200 mg/L can be accepted.

4.2. Vanadium interferes strongly, but can be tolerated up to 10 times the concentration of hexavalent chromium present.

4.3. Iron may cause a yellow color but should not affect the colorimetric measurement at 540 nm.

4.4. Reducing matter may reduce hexavalent Cr to trivalent Cr in varying amounts. No preventative measure is available at this time; however, interference is checked by a post verification spike samples.

4.4.1. The reducing/oxidizing tendency of each matrix may be determined by characterization of samples for additional analytical parameters, such as pH, ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

4.5. For waste materials containing soluble trivalent chromium concentrations greater than four times the laboratory hexavalent chromium reporting limit, the hexavalent chromium results obtained using this method may be biased high due to method-induced oxidation.

5.0. Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

6.0. Equipment and Supplies

6.1. Instrumentation

- pH meter, Orion benchtop or equivalent
- Spectrophotometer for use at 540 nm with a light path of 1 cm (Hach DR2800 or equivalent).

6.2. Supplies

- Membrane filters, 0.45 um and 0.1 um (NALGENE disposable)
- Glasswares: volumetric pipets (class A) and volumetric Flasks (class A)
- Graduated Cylinder (class A)
- Transfer pipettes
- Calibrated pipettes: Eppendorf or equivalent
- Vacuum pump
- Specimen Cups (plastic)
- Magnetic stirrer (teflon-coated)

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Sulfuric acid (10% v/v) - Measure 100 ml of concentrated reagent grade H_2SO_4 into a 1000 ml volumetric flask (1/2 filled w/D.I. water) and dilute to mark with deionized water. Reagent is stable for 6 months. Store at room temperature.

7.1.2. Indicator solution - Place 5.0 g 1, 5-Diphenylcarbazide (AR Grade) in an amber 1000ml volumetric flask and dilute to mark with acetone. Prepare monthly or when solution becomes cloudy. Store at room temperature.

7.1.3. Acetone (reagent grade) - Store at room temperature. For stability information, refer to manufacturer's instructions or 2 years from date opened if no instructions are available.

7.2. Standards

7.2.1. Primary Potassium Dichromate ($K_2Cr_2O_7$) Stock Standard (1000 mg/L Cr+6) - Weigh 0.2829 g of dried (105°C) analytical reagent grade (or better) potassium dichromate ($K_2Cr_2O_7$) crystals (J.T.Baker catalog nos. 3090, 3093 or equivalent). Dissolve in deionized water in a 100ml flask. Dilute to the mark. Standard solution is stable for 6 months. Store at room temperature.

7.2.2. Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6) - Prepare by adding 10ml of the 1000 mg/L Primary Potassium Dichromate Standard (see Section 7.2.1) to a 100ml volumetric flask and dilute to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.3. Hexavalent Chromium Primary Spiking Solution (10 mg/L Cr+6) - Prepare by adding 1.0ml of 1000 mg/L Cr+6 Standard (see Section 7.2.1) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.4. Secondary Potassium Dichromate ($K_2Cr_2O_7$) Stock Standard (1000 mg/L Cr+6) - Weigh 0.2829 g of dried (105°C) analytical reagent grade (or better) potassium dichromate ($K_2Cr_2O_7$) crystals (J.T.Baker catalog nos. 3090, 3093 or equivalent). *The source of this standard must be from a different lot or manufacturer than the Primary Potassium Dichromate Stock Standard detailed in Section 7.2*.1. Dissolve in deionized water in a 100ml flask. Dilute to the mark. Standard solution is stable for 6 months. Store at room temperature.

7.2.5. Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) - Prepare by adding 10ml of 1000 mg/L Cr+6 Secondary Standard (see Section 7.2.4) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.6. Hexavalent Chromium Secondary Spiking Solution (10 mg/L Cr+6) - Prepare by adding 1.0ml of 1000 mg/L Cr+6 Secondary Standard (see Section 7.2.4) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

8.0. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE[2]	250 mL	Cool 4 ±2°C	24 Hours	SW 7196A

[1] Samples must be analyzed within 24 hours from time of collection. Store samples at 4°C before analysis.
[2] High-density polypropylene container

9.0. Quality Control

9.1. **Sample QC** - - The following MB, LCS, and PVS are analyzed with each batch of 20 samples or each time samples are setup, whichever is more frequent. The DU is analyzed at a frequency of 1 per 10 samples or each time samples are set up, whichever is more frequent.

9.1.1. Method Blank (MB): Use deionized water for the method blank. Analyze using all reagents in the same volumes as used in the analysis of samples. The blank value should fall below the reporting limit if not, all samples associated with the method blank must be re-analyzed. Do not subtract the blank value from sample values.

9.1.2. Post Verification Spike (PS): The PVS concentration must be two times the concentration of Cr(VI) found in the sample or 30.0 ug/L (whichever is greater). If necessary, dilute the PS to fall within the range of the calibration curve. If the PS recovery is not within 85-115%, dilute the sample and re-analyze.

Note: The post spike is identified as “MS” in TALS QC.

9.1.3. Matrix Duplicate (DU): A duplicate is prepared and analyzed in exactly the same manner as the original sample. The relative percent difference (RPD) between the sample and duplicate should be less than or equal to 20%.

9.1.4. Laboratory Control Sample (LCS): The hexavalent chromium LCS is a certified QC standard purchased from Phenova (Catalog WP

Hexavalent Chromium). The LCS is used to monitor the accuracy of the entire analytical process. The LCS recovery must be within vendor specified QC limits or all samples associated with the LCS must be reanalyzed.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): The ICV (75.0 ug/L Cr (VI)) is analyzed immediately after an acceptable initial calibration. The ICV standard must be from a source separate from the calibration standards (i.e., different manufacturer or separate lot) and its recovery must be within ±10% of the expected value. The ICV is prepared by adding 0.375ml of a 10 mg/L Cr6+ Hexavalent Chromium Secondary Spiking Solution (Section 7.2.6) into a 50 ml volumetric flask and diluting to 50ml with deionized water. If the measured concentration exceeds the ±10% limit, a second analysis should be performed. If the result still exceeded the ±10% limit, the analysis should be terminated until the source of the problem is identified and corrected.

9.2.2. Calibration Check Verification (CCV): A midpoint Calibration Check Verification spiked at 75.0 ug/L is prepared in the same way as the ICV (See Section 9.2.1). The CCV is analyzed after every 10 samples (20 readings including backgrounds) and after reading the last sample. The result must be within ± 10% of the true value. If not, all samples following the last acceptable CCV must be re-analyzed.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB): Initial Continuing Calibration Blank (ICB) must be analyzed after the ICV. Continuing Calibration Blank (CCB) must be analyzed after every 10 samples or after each Calibration Check Verification (CCV). Use deionized water for the ICB/CCB. The value of the ICB/CCB should be less than the RL. If not, all samples following the last acceptable ICB/CCB must be re-analyzed.

9.2.4. Minimum Reporting Level (MRL), 10ppb: The MRL must be analyzed daily before samples are analyzed. Prepare the MRL by adding 0.05ml of a 10 mg/L Hexavalent Chromium Primary Spiking Solution (Section 7.2.3) into a 50 ml volumetric flask and diluting to 50ml with deionized water. The results must fall within 50-150% of the true value.

10.0 Procedure

10.1. Calibration

10.1.1. Prepare fresh calibration standards everyday before analysis.

10.1.2. Initial Calibration Standards: (6 points). Place about 25 mL of deionized (DI) water in six 50 ml flasks. Prepare the six points as follows:

Cr(6+) Standard conc. (ug/L)	Volume (ml) of 10 mg/L Cr+6 (Section 7.2.3) to add	Final volume (ml) using Deionized water

0.0	0.0	50
10.0	0.05	50
50.0	0.25	50
75.0	0.375	50
100	0.50	50
200	1.0	50

10.1.3. Analyze the calibration standards in the manner detailed in Sections 10.2.1 thru 10.2.7. Zero the spectrometer with the 0 ug/L standard.

10.1.4. Perform linear regression on data resulting from analysis of the initial calibration. The correlation coefficient must be 0.995.

10.1.5. Analyze the Initial Calibration Verification (ICV) standard in the same manner as the initial calibration standards detailed above. If the ICV meets the criteria listed in Section 9.2.1, the analyst may continue with sample analysis (Section 10.2).

10.2. Sample Analysis

10.2.1. Warm up spectrophotometer and set to 540 nm.

10.2.2. Place 45.0 mL of sample into a 100 ml specimen cup. Also, place 45 mL of sample in a separate cup for background.

10.2.3. Add 1.0 ml indicator solution (see Section 7.1.2) and swirl. (Do not add indicator to background sample).

10.2.4. Slowly add 10% H_2SO_4 (See Section 7.1.1) dropwise to sample and background while measuring pH until pH is 1.5-2.5. Record reading.

10.2.5. Place sample in a 50 ml volumetric flask and bring to volume with deionized water. Pour back into original cup and swirl.

10.2.6. Let stand 5-10 minutes for full color development.

10.2.7. Read calibration standards, QC samples, and samples on spectrophotometer at 540 nm. Analyze the calibration check standard and calibration check blank immediately following the calibration standards.

10.2.8. Dilute samples that exceed calibration curve using deionized water. Recolorize and read again on spectrophotometer noting dilution factor.

10.2.9. Continue analyzing samples and quality control samples by first reading the sample followed by its background sample.

10.2.10. After every 10 samples and after reading the last sample in the batch, verify the calibration by reading Calibration Check Verification (CCV) and Continuing Calibration Blank (CCB).

11. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculate the hexavalent chromium result for each aqueous sample as follows:

Hexavalent Chromium in ug/L = A x E

A = Concentration from the calibration curve in ug/L
E = Dilution (if necessary)

11.4. Trivalent Chromium Calculation: When required, the calculation for trivalent chromium is completed using the TALS method '7196a_CR3' which must be logged for the samples being processed along with methods for total chromium (metals) and hexavalent chromium. Upon completion of the total and hexavalent chromium analyses the analyst may process the trivalent chromium results as follows:

- To have TALs perform an automated trivalent chromium calculation: on the trivalent chromium batch information page enter '1' (for 'yes') in response to the prompt to automatically perform the calculation. As long as both methods are at 2nd level review status in TALS the program will automatically pull in the metals results for chromium and the hexavalent chromium results to perform the automated calculation. TALS will display an 'ok' message on the worksheet tab if the Cr6 result is less than Total Cr result.

- If the analyst intends to manually enter trivalent chromium results an entry of '0' (for 'no') should be made the and the results entered manually.

11.5. Data Reduction

11.5.1. Record analytical data directly into TALS and in the 'Hexachrome Run Logbook.'

11.5.2. Record Reagent information on the "batch information" page.

11.5.3. Special comments or observations should be recorded on the "worksheet" tab. Record Sample absorbances and pH readings on the "worksheet" tab.

11.5.4. Attach the calibration curve plot and the logbook pages for the analysis to the analytical batch as pdf files.

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.5.5.1 Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2 Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3 Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.4 Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

12. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are generated as a result of this analysis:

- Water Retain Samples -These materials are originally received aqueous samples. These solutions are then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9 with sodium bicarbonate (Siedler Chemical SC-0219-25). This solution is discharged into the municipal sewer system.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 7196A.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

N/A

18.0. Revision History

- Revision 11, 10/01/2021
  - Sec. 9.2.4: Added criteria to analyze a Minimum Reporting Level (MRL) daily.
- Revision 10, 14 May 2020
  - Updated SOP header with Eurofins logo.
  - Sec 1.1: Clarified matrix applicability for this SOP.
  - Sec. 9.1: Revised the frequency of Matrix Duplicate from 1/20 samples to 1/10 samples.
- Revision 9, 12 February 2019
  - Sec. 9.1.4: Changed LCS source vendor from ERA to Phenova.
  - Sec. 11.5.5: Replaced Wetchemistry Data review checklist (EDS-WI-008) to TALS Data Review Checker (DRC); instructions added.
  - Deleted Sec 15.6, WI EDS-WI-008, not applicable; subsequent sections adjusted accordingly.
- Revision 8, 30 Nov 2016
  - Sec. 11.4: Added Cr3 calc procedure
- Revision 7, 26 November 2012
  - Section 9.1.2: Revised the minimum concentration for PS from 15 ug/L to 30 ug/L to comply with Method 7196A.
- Revision 6, 04 October 2012
  - Section 1&12: Updated LQM reference to reflect the most current LQM revision.
  - Sec 5: Updated to comply with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP)
  - Sec. 9.1.1: Revised to include corrective action procedure when MB is greater than the RL.
  - Sec. 9.1.4: Changed limits from 85-115% to vendor specified QC limits.
  - Sec. 9.2.2 & 9.2.3: Revised to include corrective action procedure when CCV and CCB are outside the acceptance limits.
- Revision 5, 7 September 2010
  - Section 2.0: Removed text 'Dissolved;' this SOP is only applicable to aqueous samples for method SW7196A which are not filtered.
  - Sec 3: Revised to update LQM reference for the list of definitions.
  - Section 6.2: Added graduated cylinders (class A) and transfer pipettes to the list of supplies.
  - Removed Nitric acid on list of reagents, not applicable for the analysis of Cr6+ waters.

- Section 9.1.1: Revised method blank criteria from <MDL to <RL.
- Section 9.1.4: Replaced LCS vendor specified limits to 85-115%.
- Section 9.2.2: Removed text: “immediately after analyzing the calibration standards.” Renamed CCS to CCV.
- Section 9.2.3: Revised CCB criteria from <MDL to <RL.
- Section 10.1.4: Revised the correlation coefficient criteria from ≥0.997 to ≥0.995.
- Section 13 & 14: Revised to comply with the current Method SOP format (CW-QS-002, Writing a Standard Operating Procedure (SOP)).
- Section 11.4: Revised Data Reduction section in accordance with TALS.
- Section 15: Added applicable references.

- Revision 4, January 22, 2008

  - Updated to new format as per TestAmerica’s SOP format.
  - Added the shelf-life and storage requirements to reagents in section 7, Reagents and standards.
  - Added J.T.Baker (Catalog Nos. 3090, 3093 or equivalent) as source of primary and secondary standards. Deleted reference to Inorganic Ventures as a source of the primary standard.
  - Replaced the source standard used for the preparation of 10 mg/L Cr+6 (primary and secondary spiking solution) from 100 mg/L Cr+6 to 1000 mg/L Cr+6.
  - Added the control limits for Post Verification Spike (PVS).
  - Added ICV as Instrument QC.
  - Added the preparation procedure and control limits for the Calibration Check Standard (CCS).
  - Deleted Matrix spike (matrix spike low and matrix spike high) as sample QC requirement.
  - Section 10.3.3: Revised pH range from 1.6-2.2 to 1.5-2.5 to reflect method requirements.
  - Standardized all analytical units to ug/L and mg/L.
  - Added the following to the References section: TestAmerica Edison SOP ED-GEN-022 (Training) and TestAmerica Laboratory Quality Manual (ED-QA-LQM).

Title: Mercury Analysis for Water and Wastewater using EPA 245.1 and SW846 7470A; Mercury in Drinking Water using EPA 245.1; Leeman Mercury Analyzer (Cold Vapor Technique)

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

EPA Method 245.1 and SW846 Method 7470A are applicable to the determination of mercury in water matrices. Mercury may be found in water in both inorganic and organic forms. Organomercury compounds must first be broken down to respond to the cold vapor atomic absorption technique.

The typical detection limit using a 30 ml sample size is 0.2ug/L Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A digested sample is analyzed using cold vapor atomic absorption. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 The addition of potassium persulfate during the digestion step can eliminate the possible interference from sulfide in the sample without affecting the recovery of inorganic mercury.

4.2 Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/l total copper.

4.3 Samples that contain high levels of chloride have a potential to interfere due to a reaction that takes place during the oxidation step. During this step chloride is converted to free chlorine which absorbs light at 253.7 nm. The analyst must not allow the chlorine to be swept into the optical cell. The possibility of the chlorine interfering with the analysis can be minimized by using an excess of up to 7.5 ml hydroxylamine sulfate.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
a)Mercury b)(1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/M^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1. Leeman Laboratories Inc. Hydra II AA Automated Hg Analyzer

6.1.2. Computer and Monitor with Leeman Envoy software

6.1.3. Block digestor (Environmental Express or SCP Science): Adjustable and capable of maintaining a temperature of 90 -95°C.

6.2 Supplies

6.2.1. 50 ml Hot Block Digestion Cups

6.2.2. 100 ml graduated cylinder

6.2.3. Eppendorf Pipettes and tips in various sizes

6.2.4. 100 ml volumetric flasks

6.2.5. 15 ml sample cups

6.2.6. 10 liter carboy container

6.2.7. Pump tubing:

- Sample, viton, blue tab
- Reductant, red tab
- Drain, blue tab
- Rinse, black tab

6.2.8. Drying Tube – Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.

6.2.9. Nitrogen or Argon supply - capable of producing 80 PSI.

7. Reagents and Standards

7.1. Reagents

Storage requirements: store at room temperature

Life of Reagent: - Concentrated acids: refer to manufacturer's instructions
- Laboratory prepared reagents and diluted acids: one year

Document prepared reagents in the Reagent module located in the TestAmerica Laboratory System (TALS).

7.1.1 Sulfuric acid - Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.2 Nitric acid - Concentrated (Trace Grade or Equivalent)

7.1.3 Hydrochloric acid-Concentrated (Trace Grade or Equivalent)

7.1.4 Potassium Permanganate (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.5 Sodium Chloride (analytical reagent grade); for stability information, refer to manufacturer's instructions.

7.1.6 Hydroxylamine Hydrochloride (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.7 Stannous Chloride (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.8 Potassium Persulfate (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.9 Deionized water - 18 megohm minimum

7.1.10 10% Hydrochloric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 800 ml of concentrated HCl and bring the final volume up to 8 liters with deionized water.

7.1.11 10% Stannous chloride solution - Add 50 g of $SnCl_2$ to 500 ml 10% HCl solution.

7.1.12 Sodium chloride/Hydroxylamine Hydrochloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water.

7.1.13 Potassium Permanganate ($KMnO_4$) 5% solution w/v - Dissolve 100 g of $KMnO_4$ in deionized water and dilute to 2 liters using deionized water.

7.1.14 0.15% Nitric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 12mL of concentrated HNO_3 and bring the final volume up to 8 liters with deionized water.

7.1.15 Potassium Persulfate ($K_2O_8S_2$) 5% solution w/v: Dissolve 50 g of potassium persulfate in 1 liter of deionized water.

7.2 Standards

Storage requirements: all standards are stored at room temperature
Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – made fresh daily
Working standards – made fresh daily

(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: see Attachment 1 for example certificates of analysis (COA) for all of the standards mixes listed below. The COA lists the manufacturer's part number, certified concentration and shelf life.

Document standard preparation in TALS (see Section 11.5.2).

7.2.1 Stock Mercury Calibration (10 ppm Hg) - Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.2. Stock Mercury Quality Control Standard (10 ppm Hg) - Purchase from Inorganic Ventures; store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard must be purchased from a second source vendor.

7.2.3. Intermediate Calibration Standard (DCAL-Int): Dilute 1 ml of Stock Mercury Calibration (Sec 7.2.1) to 100 ml with 0.15% HNO_3. The resulting solution will contain 100ppb Hg.

7.2.4. Intermediate Quality Control Standard (DQCS-Int): Dilute 1 ml of Stock Mercury Calibration Verification Standard (Sec 7.2.2) solution to 100 ml with 0.15% HNO_3. The resulting solution will contain 100ppb Hg.

7.2.5. Calibration Standard Preparation: Use six 50 ml hot block digestion cups to prepare the standards. Add small portion of 0.15% HNO_3 to each cup. Working in increasing order, spike the appropriate cup with 0.0, 0.06, 0.3, 0.6, 1.5, and 3.0 ml of working solution DCAL-Int (Sec 7.2.3). Bring to final volume of 30 ml and mix thoroughly. The corresponding concentrations are 0.0ppb, 0.2ppb, 1.0ppb, 2.0ppb, 5.0ppb, and 10.0ppb mercury respectively. For drinking water analysis, the 2.0ppb standard is also analyzed as the Maximum Contaminant Level (MCL) standard

7.2.6. Quality Control Standard (QCS) Preparation: Add a small portion of 0.15% HNO_3 to a 30 ml hot block digestion cup and spike 1.5 ml of DQCS-Int (Sec 7.2.4). Bring up to final volume and mix thoroughly. The resulting solution will contain 5.0 ppb of Hg.

7.2.7. Initial Calibration Verification (ICV) standard: Add a small portion of 0.15% HNO3 to a 30 ml hot block digestion cup and spike 1.5 ml of DQCS-Int (Sec 7.2.4). Bring up to final volume and mix thoroughly. The resulting solution will contain 5.0 ppb of Hg.

7.2.8. Continuing Calibration Verification (CCV) standard 5.0ppb: The 5.0 ppb

prepared from the calibration standards (Sec 7.2.5) is used as the Continuing Calibration Verification standard (CCV).

7.2.9. Maximum Contaminant Level (MCL) standard 2.0 ppb *for drinking water analysis:* The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard.

7.2.10. Reporting limit check standard (CRI) 0.2 ppb: The 0.2ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the CRI standard. This standard is being analyzed as part of some client's special projects.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [2]	Reference
Waters	P. FP, G[1]	250 ml	HNO_3, pH < 2 prior to shipment; if not, acidify upon receipt in lab; Cool 4±2°C	28 Days	40 CFR Part 136.3

[1] Polyethylene, fluoropolymer, glass
[2] Inclusive of digestion and analysis
[3] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 16 hours for method 245.1 drinking water and 24 hours for non-potable water. Just prior to digestion or direct analysis, verify pH<2. If pH≥2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	**245.1**: < RL or; <10%sample concentration (whichever is greater) **7470A**: < RL; **or;** < 5% of the reg limit; **or,** < 5% of the measured sample concentration (whichever is greater)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	**245.1**: 85-115% **7470A**: 80-120%
Matrix Duplicate (DUP)[1]	1 in 20 or fewer samples	20% RPD
Matrix Spike (MS)[1]	**245.1**: 1, if 10 samples or less; 2 if 11-20 samples **7470A**: 1 in 20 or fewer samples	**245.1**: 70-130% **7470A**: 75-125%
Matrix Spike Duplicate (MSD)	When requested by client	**245.1**: 70-130% **7470A**: 75-125% **For both 245.1** & **7470A**: If MS and MSD are both ≥ 5X CRDL, then 20% RPD. If MS and MSD are less than the CRDL, the RPD is not calculated; otherwise ±CRDL.
Serial Dilution (SD) for 7470A	1 in 20 or fewer samples	±10%

[1] The sample for DUP and MS are randomly selected, unless specifically requested by a client. Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.

9.1.1 **Method Blank:** One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. If the Mercury concentration in the MB is above the specified control limit, the batch must be prepared again and the samples reanalyzed.

- For 7470A, results must be less than RL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample, whichever is greater.
- For 245.1, results must be less than RL or less than 10% of the determined Mercury concentration in the sample, whichever is greater.

9.1.2 **Laboratory Control Sample (LCS):** A laboratory control sample must be analyzed with each batch of samples digested. A blank is spiked with 0.1

ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. Results of the aqueous LCS must fall within ±15% of the true value for Method 245.1 and ±20% for Method 7470A.

9.1.3 **Matrix Duplicate (DUP):** A duplicate is analyzed for each batch of samples digested. If original sample and duplicate are both ≥ CRDL, then 20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.4 **Matrix Spike (MS):** A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. A recovery of 70-130% for Method 245.1 and 75-125% for Method 7470A is required.

9.1.5 **Matrix Spike Duplicate (MSD):** When requested by the client, a matrix spike duplicate is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. A recovery of 70-130% for Method 245.1 and 75-125% for Method 7470A is required. For both 245.1 & 7470A: if original sample and duplicate are both ≥ CRDL, then 20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.6 **Serial Dilution (SD):** For method 7470A, a five fold serial dilution must be performed on one sample per batch. The sample should contain a sufficiently high concentration; minimally a factor of 25 times above the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. Results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

9.2 Instrument QC

9.2.1. **Initial Calibration Verification (ICV):** Initial calibration is verified after calibration. The ICV solution should be prepared using a second source vendor; see section 7.2.7 for preparation instructions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the ICV; see Sec 7.2.7 for preparation instructions. For 245.1, the results must not differ from the true value by more than 5%. For 7470A, the results must be within 10% of the true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.2. **Continuing Calibration Verification (CCV):** Calibration verification is performed after the calibration, after every 10 samples, and at the end of the run. The CCV solution should be prepared from the same CAL

standard as used to prepare the calibration solutions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the CCV.

For 245.1 and 7470A, CCV must not differ from the true value by more than 10%. If it does, stop the analysis and recalibrate. Re-analyze the previous ten samples following the last good calibration verification.

9.2.3. **Initial and Continuing Calibration Blank (ICB/CCB):** ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. For methods 245.1 and 7470A, the absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

9.2.4. **Maximum Contaminant Level (MCL):** For drinking water analysis, one MCL standard shall be analyzed per calibration. The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard. The result must be within 50% of the true value. If it's outside of the acceptable limit, terminate the analysis, correct the problem, and recalibrate the instrument.

9.2.5. **Quality Control Standard (QCS):** The calibration is verified after calibration using a second source vendor; see Sec 7.2.6 for preparation instructions. For 245.1 and 7470A, the results must not differ from the true value by more than 10%. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.6. **Reporting limit check standard (CRI):** At the beginning of the analysis, verify the accuracy at the reporting limit (RL) by analyzing a solution at the RL level. RL check solution is analyzed to demonstrate that the mercury analyzer is capable of detecting the target analyte at the reporting limit (RL). Laboratory limits are 50-150% of the true value. This standard is being analyzed as part of some client's special projects.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Filtration Procedure for Dissolved Mercury not filtered in the Field

10.1.1.1 The unpreserved sample must be filtered through a 0.45um filter unit as soon as practical after collection.
10.1.1.2 Collect the required volume of filtrate by using a 0.45um filter unit and a vacuum pump.
10.1.1.3 Acidify the filtrate with 1:1 HNO_3 to a pH of <2.
10.1.1.4 The method blank (MB) must be filtered and digested under the

same conditions as the lab filtered samples.

10.1.2 If sample is preserved in laboratory, hold sample for 16 hours for method 245.1 drinking water and 24 hours for wastewater following acidification. Verify and record if sample pH is < 2.0 after 16 or 24 hours (depending on the analysis- drinking water or wastewater) and prior to digestion/analysis. If pH is > 2.0, add HNO3, hold for 24 hours and verify pH.

10.1.3 Transfer 30 ml sample (DI water for MB and LCS) or standard, or an aliquot diluted to 30 ml, to an appropriately identified 50 ml hot block digestion cup. For QA samples, transfer 3 aliquots of 30 ml sample to three digestion cups labeled as SAMPLE, DUP and MS. Spike LCSW and MS with 0.1 µg of mercury (0.3 ml of DCAL-INT standard).

10.1.3.1. Due to insufficient sample volume or an unusual matrix (e.g., samples with potentially high interferences), it may be necessary to perform a pre-digestion dilution. For example, a 10X pre-digestion dilution can be performed by transferring 3 mL of sample to 50 mL hotblock cup. Add 27 ml of deionized water. Record 3 mL initial volume and 30 mL final volume in TALS (see Sec 11.5.2). Continue to section 10.1.3.

10.1.4 Add 1.5 ml concentrated H_2SO_4 and 0.75 ml concentrated HNO_3 mixing well after each addition.

10.1.5 Add 4.5 ml of potassium permanganate solution to each bottle. Mix well and let stand for 15 minutes (minimum); if the color is still present, then proceed to section 10.1.6; if the color has disappeared, then see section 10.1.5.1.

10.1.5.1 Discard the aliquot. Using a new 50mL hot block digestion cup, dilute the sample so that the final volume is 30mL. For example, for a 3X dilution: add 20mL of 0.15 Nitric Acid (sec 7.1.14), 10mL of sample, and mix. Record the initial and final volumes in TALS. Add 4.5mL of potassium permanganate as described in sec 10.1.5 and wait 15min. If purple color is still present, proceed to sec 10.1.6; if not, a larger dilution is necessary.

10.1.6 Add 2.4 ml potassium persulfate solution to each bottle.

10.1.7 Heat for 2 hours in a 95° C hot block digestor. Remove from block digestor and cool.

10.1.8 Add 1.8 ml Sodium chloride - Hydroxylamine hydrochloride solution to reduce the excess permanganate. Mix well; solution should become colorless. If necessary additional Sodium chloride - Hydroxylamine HCl solution may be added.

10.1.9 Due to differing rates of evaporation, the final volume of the standards/samples maybe different. The standards and samples, including all QC, must be at the same final volume before analysis; if not, find the sample or standard with the highest final volume (e.g., 41 mL) and add deionized water to any sample or standard to bring them to the same final volume . Mix well. Wait at least 30 seconds after decolorization before analyzing.

10.2 Calibration

10.2.1. The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in TALS (see Sec 11.5.2).

10.2.2. Prepare five calibration standards, blank and calibration verification standard as detailed in sections 7.2.5 and 7.2.6.

10.2.3. Calibration Curve Read-Back:

10.2.1.1 Low-Level Readback (at the RL) – evaluate the 0.2 ug/L calibration standard. The %RE (relative error) must be +/- 20% of the true value (see Sec 11.4). If %RE is outside of criteria limits, stop the analysis and recalibrate.

10.2.1.2 Mid- Level Readback – evaluate the 5.0 ug/L calibration standard (the mid-level calibration standard). The %RE (relative error) must be +/- 10% of the true value (see Sec 11.4). If %RE is outside of criteria limits, stop the analysis and recalibrate.

10.3 Sample Analysis

10.3.1 Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.

10.3.2 The following analytical run sequence is currently used:

Instrument Calibration (Blank and five standards)

ICV
ICB

CRI
QCS
MCL (for drinking water analysis)
CCV
CCB
10 Samples
CCV
CCB
Repeat until run is completed
CCV
CCB

10.3.3 Instrument Operation:

10.3.3.1. Turn on Computer, Monitor and the Hg analyzer (Hydra IIAA).

10.3.3.2. Plumbing the Reagent Lines:

10.3.3.2.1. One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.

10.3.3.2.2. Reductant (Red): Connect tab end of tube to the reductant tubing that is connected to the reductant bottle and the other end to the mixing tee.

10.3.3.2.3. Sample (Blue): Connect tab end of tube to the autosampler probe and the other end to the mixing tee.

10.3.3.2.4. Drain (Blue): Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.

10.3.3.2.5. Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.

10.3.3.3. Preparation of Reagents:

10.3.3.3.1. Pour the 10% $SnCl_2$ solution into the reductant bottle.

10.3.3.3.2. Pour the 10% HCl solution into the rinse reservoir bottle.

10.3.3.4. Start the Program:

10.3.3.4.1. Click the Envoy icon on the computer desktop
10.3.3.4.2. Click Method, select 245.1_7470A, and select OK
10.3.3.4.3. On the main screen, click the StartUp icon. Wait 15 minutes before analyzing calibrating.
10.3.3.4.4. Click Sequence Tab on bottom of screen
10.3.3.4.5. Click Sequence on top of screen
10.3.3.4.6. Select Open, then select New
10.3.3.4.7. Using the Prep Batch Sheet and hand scanner, enter the sample barcodes into the Sample ID column. Include the Serial Dilution and all needed sample dilutions
10.3.3.4.8. Pour out the digested calibration standards and samples into the proper locations on the autosampler.
10.3.3.4.9. Click the Run Sequence icon to begin calibration and to run samples.

10.3.3.4.10. To shut down the instrument, click the Stop icon on the main screen.

11.0 Calculations / Data Reduction

11.1 Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation in aqueous samples :

$$\text{Concentration = mg/ L} = \frac{C \times V1 \times D}{V2}$$

Where:
C= Element concentration from instrument (ppb)

V1= Final volume of sample digested (in liters)
D= Dilution performed on sample

V2= Initial volume of sample digested (in liters).

11.4. Relative Error (%RE):

% Relative Error = (100) (MC-TC)/TC

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Processing:

11.5.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.5.2. Record standard/sample preparations in the Analyst Desktop II program located in the TestAmerica Laboratory System (TALS) Reagent module. The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation and expiration dates.

11.5.3. Record the following reagents and the volume used for sample preparation in the batch information page under "batch comments" in TALS Analyst Desktop II: concentrated sulfuric acid, concentrated nitric acid, potassium permanganate, potassium persulfate and sodium chloride-hydroxylamine hydrochloride. Record reagents in TALS by opening the prep batch, click on "edit" and then right click to choose "view batch information." Enter the information in the "batch comments" section.

11.5.4. Import Data to TALS

Click the Analysis tab

11.5.4.1. Select the analytical run that needs to be imported
11.5.4.2. Select Statistics
11.5.4.3. Click Load and select TALS Import, click OK
11.5.4.4. Click Report, click CSV File
11.5.4.5. Name the import file (e.g., batch name, today's date)
11.5.4.6. The newly created import file is in the Import Folder on the desktop Send to TALS Import Folder

11.5.5. Creating the Raw Data PDF:

11.5.5.1. Click the Analysis Tab, select Detailed

11.5.5.2. Click Load, select Use This To Make PDF

11.5.5.3. Click Report, Click Printer

11.5.5.4. Include the calibration curve graph:

11.5.5.4.1. Go to Methods Tab and click Calibration, click Print

11.5.5.5. Combine both documents using the PDF Creator program. The new document is located in the Documents folder on the C:\ drive. Add this document to the "Doc's" location in the analytical batch.

11.5.6. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch.

11.5.6.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.6.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.6.3. Acknowledge by filling in responses to all unacknowledged findings.

11.5.6.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.6.3.2. Fill in appropriate comments in the response box, then hit 'OK.'

11.5.6.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer

12. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Instrument Detection Limit:

The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. For 245.1, the IDL is determined by multiplying the average of the standard deviations obtained from the analysis of 10 reagent blanks by 3. For 7470A, the IDL is determined by multiplying the average of the standard deviations obtained from the analysis of 7 reagent blanks by 3.14

12.3 Linear Dynamic Range (LDR)

The upper limit of the LDR must be established. It must be determined from a linear calibration prepared from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. The LDR should be determined by analyzing succeedingly higher standard concentrations of mercury until the observed analyte concentration is no more than 10% below the stated concentration of the standard. The determined LDR must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDR should be verified annually or whenever, in the judgement of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

12.4 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.5 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

12.6 Lower Limit of Quantitation (LLOQ)

12.6.1. LLOQ verification: prior to analyzing samples under method 7470A, after any change that may affect the LLOQ, and quarterly, the LLOQ must be verified. The LLOQ is initially verified by the analysis of 7 replicate samples spiked at the LLOQ (0.2 ppb). The seven replicates must be digested prior to analysis. The LLOQ is verified when the mean recovery of the 7 replicates is +/-30% of the true value and the RSD < 20%.

12.6.2. LLOQ on-going verification: the LLOQ is re-verified quarterly by the analysis of a method blank (MB) and 1 blank sample (deionized water spiked at the LLOQ (0.2 ppb). The MB and LLOQ samples must be digested prior to analysis. The LLOQ is re-verified if the MB result is +/- 0.5 times the reporting limit for each analyte and when the recovery of the LLOQ sample is +/-30% of the .true value.

13. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out.

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

15. References / Cross-References

15.1. Determination of Mercury in Water by Cold Atomic Absorption Spectrometry, EMSL-Cincinnati, EPA/600/R-94/11, May 1994; Method 245.1 Revision 3.0.

15.2. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.

15.7. Leeman Hydra II AA Operating Manual

16. Method Modifications:

Item	Method No.	Modification
Sample Preparation	SW 7470A	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's improvement that will reduce error due to loss of Mercury.
Sample Preparation	SW 7470A EPA 245.1	The hotblock has replaced the hot-water bath for digestion. This modification has been made to reduce cross-contamination (the hotblock tubes are disposable).
Sample Preparation	SW 7470A EPA 245.1	The typical prep sample size is reduced to 30ml (previously 100ml). This modification was made to allow for limited available sample volumes. Reagent volumes were adjusted to maintain sample to reagent volume ratio
Sample Preparation	EPA 245.1	The Initial Calibration Verification (ICV) will be prepared from a secondary source (as required in Method SW7470A), rather than the calibration standard source. Method 7470A and 245.1 will be prepared and analyzed in the same batch which improves efficiency and reduces waste. CCV will be prepared using the same source as the calibration standards.
Sample Preparation	SW 7470A EPA 245.1	When KMnO4 is added to the sample and the purple color is not present in the sample in the 15 minute period, the aliquot is discarded and a dilution is preformed using a lesser amount of sample. The purple color is checked after 15 minutes and the reporting limit is adjusted to reflect the dilution.

17. Attachments

Attachment 1: Example Certificate of analysis (10 ppm Hg)

18. Revision History

- Revision 15, dated 20 September 2023
  - Sec 10.1.5: Revised to remove instructions for adding more KMnO4 to all samples and standards when purple color persist after 15 minutes of addition.
  - Sec 10.1.5.1: Added sample dilution instructions for a sample that doesn't maintain the purple color for 15 minutes.
  - Sec 16: Added method modification notation for samples that don't maintain the purple color after 15 minutes.

- Revision 14, dated 11 August 2022
  - Sec 12.6: Added the lower limit of quantitation (LLOQ), associated instructions and recovery limits.

- Revision 13, dated 13 September 2021
  - Sec 7.2.10 & 9.2.6: Revised to clarify use of CRI standard.

- Revision 12, dated 03 August 2020
  - Updated header with Eurofins logo.
  - Sec 6.1.1: Replaced Hydra AA with Hydra IIAA
  - Sec 6.1.2: Updated Hg software from WinHg to Envoy.
  - Sec 8.0: Revised the required sample volume from 500ml to 250ml.
  - Sec 9.1: Revised MB control limits for 245.1 and 7470A to <RL; added MSD to the QC table.
  - Sec 9.1.1: Updated the control criteria for 245.1 and 7470A from <MDL to <RL.
  - Sec 9.1.3: Updated the criteria for the matrix duplicate to reflect current laboratory practices.
  - Sec 9.1.5: Added MSD to the list of Sample QC.
  - Sec 9.2.2: Revised the CCV recovery limit for method 7470A to 10%.
  - Sec 10.2.3: Added calibration curve read-back criteria.
  - Sec 10.3.3.4: Updated instructions for the instrument/software operation.
  - Sec 11.4: Added Relative % error calculation.
  - Sec 11.5.2: Added reagent/standard information to document in TALS ADII.
  - Sec 11.5.3.: Added requirements to record reagent volumes in the batch information page of TALS ADII; subsequent sections adjusted accordingly.
  - Sec 11.5.4 & 11.5.5: Updated instructions for data import and raw data pdf generation.
  - Sec 11.5.6: Added Data Review Checker (DRC) instructions.
  - Sec 15.7 & 15.8: Deleted work instructions EDS-WI-007 (TestAmerica Edison Metals Data Review Checklist) and WI EDS-WI-125 (TestAmerica Edison Metals Initial-Calibration Data Review Mercury checklist), not applicable.

- Revision 11, dated 25 April 2018
  - Sec 8: Added footnote 3 to clarify instructions for metals sample preservation (verify pH <2.0 prior to digestion).
  - Sec 10.1.2: Added procedure for samples which pH are >2.0 and acid preservation is performed in laboratory.
  - Sec 12.3: Added procedure for the determination of linear dynamic range.

- Revision 10, dated 05 February 2018
  - Sec 7.2.10: Added CRI to the list of standards.
  - Sec 9.2.6: Added CRI to the list of instrument QC.
  - Sec 10.3.2: Revised the instrument sequence list to include CRI.

- Revision 9, dated 09 December 2015
  - Sec 7.2.7: Revised preparation procedure for ICV, standard solution should be obtained from a source different from the calibration standard. Removed reference to CCV.
  - Sec 7.2.8: Added CCV preparation procedure; standard solution same source as the calibration standard. Subsequent sections adjusted accordingly.
  - Sect 9.2.1: Revised - ICV solution should be prepared from a separate source.
  - Section 16: Added Method Modifications for using the secondary source when preparing the ICV standard for method EPA 245.1.

- Revision 8, dated 13 January 2014
  - Sec 7.1.8: Added the stock standard Potassium Persulfate to the list of reagents; subsequent sections adjusted accordingly.
  - Sect 7.1.15: Added preparation instructions for the 5% Potassium Persulfate solution.
  - Sec 7.1: Revised to include recording of reagent prep information in TALS and discontinue the use of Mercury Prep Logbook.
  - Sec 7.2.5 & 7.2.6: Revised the spiking amount to add when preparing calibration standards in 30 ml final volume; also replaced the 100 ml flasks with 50 ml digestion cups.
  - Sec 9.0: The matrix spike (MS) frequency for Method 245.1 has been revised to 1 per 10 samples to comply with the method.
  - Sec 11.4.4: Added the Mercury calibration checklist (EDS-WI-125).
  - Sec 15.8: Added WI# ED-WI-125 on the list of references; subsequent sections adjusted accordingly.
  - Sec 17: Updated the attached COA for the Hg standards.

- Revision 7, dated 02 December 2011
  - Sec 1.1: Revised detection limit to 0.2 ug/L Hg to reflect actual laboratory limits.
  - Sec 1 & 12: Revised the LQM reference for DOC and Test Methods and Method Validation to Section 19.
  - Sec 3: Revised LQM reference for the definitions.
  - Sec 5: Revised in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002, Writing a Standard Operating Procedure (SOP), most current revision.
  - Sec 7.1: Added requirements that all prepared reagents are recorded in TestAmerica Edison Mercury Std Prep Log.
  - Sec 7.2 & 10.2.1: Revised the documentation of standard preparation from logbook to TALS.
  - Sec 7.2.5: Replaced cal std 0.1 ug/L (Hg) with 0.2 ug/L.
  - Sec 9.1 and 9.1.1: Expanded MB control limit for 245.1 to reflect method's criteria.
  - Replaced PB (preparation blank) with MB (method blank) in various sections where applicable.

- Sec 10.1.1: Added procedure for filtering dissolved samples in the lab.
- Sec 10.1.2.1: Added procedure for pre-digestion dilutions.
- Sec 10.1.8: Added procedure for visually checking and adjusting final volumes for all standards and samples.
- Sec 10.3.3.11.7: Added procedure for post-digestion dilutions.

- Revision 6, dated 03 September 2009

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Combined SOP ED-MT-014 and ED-MT-015 with SOP ED-MT-017; retired SOP ED-MT-014 and ED-MT-015 at the effective date of this SOP.
  - Sec 6.1.3: Replaced water bath with block digestor.
  - Sec 6.2.1: Replaced 300 ml BOD bottles with 50 ml hot block digestion cups
  - Sec 6.2.3. Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines
  - Sec 7.1: Added 0.15% HNO3 to list of reagents. Deleted Potassium Persulfate, Magnesium Perchlorate, 0.5N H2SO4 & 5% Potassium Persulfate solution to list of reagents; Reagents deleted are not applicable to this method.
  - Sec 7.2 Standards: Revised the Hg stock standard concentrations and preparation of standards.
  - Sec 7.1.9 & 7.1.13: Revised preparation procedure to reflect actual lab practices.
  - Sec 7.2.6: Renamed the second source standard 'Initial Calibration Verification standard' to 'Quality control standard.' QCS is added in the Instrument QC and analytical run sequence.
  - Section 8: Updated the section into Table format and have included the sample container, sample size requirements and method reference.
  - Changed the verification standard concentration of ICV (3ppb) to 5ppb; CCV concentration remains 5ppb.
  - Revised control limits to comply with Method 245.1
  - Sec 9.1.1: Clarified QC limits for the Preparation Blank
  - Sec 9.1.2: Revised the LCS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±15%.
  - Sec 9.1.4: Revised the MS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±30%.
  - Sec 9.1.5: Added Serial Dilution (L) in Sample QC
  - Sec 9.2.1: Clarified the recovery limits of ICV for method 245.1; % Rec limits were revised from 10% to 5% to reflect actual laboratory practices.
  - Sec 10.3.2: Added MCL in the analytical run sequence.
  - Sec 10.3.3.2: Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines.
  - Sample size reduced from 100 ml to 30ml; preparation of the LCS (Sec 9.1.2) and MS (Sec 9.1.4) were revised to reflect this change in sample volume.
  - Sec 10.1: Adjusted reagent volume to maintain sample to reagent ratios.
  - Sec 11: Updated data processing in accordance with the new TALS.
  - Sec 15: Added applicable references.
  - Sec 16: Described the elimination of stannous chloride in the sample preparation and replacement of hot water bath as a method modification.

Attachment 1

SCP SCIENCE
Providing Innovative Solutions to Analytical Chemists

ACCREDITED

Certificate of Analysis

1.0 DESCRIPTION : ***Plasma*** **CAL – Custom Standard**

Catalogue Number : **141-110-11X**
Lot Number : **S130418021**
Matrix: 10.0% HNO3
Expiration Date : **July 2014**

2691783
ID: ME_Hg_1st_00013
Exp: 07/01/14 Post DLE
Mercury Calibration Stand

2.0 CERTIFIED VALUES AND ASSOCIATED UNCERTAINTY:

Method of Analysis: Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES)

Traceability: Applicable NIST Standard Reference Materials (see list below):

3101a	Al	3109a	Ca	3117a	Eu	3126a	Fe	3134	Mo	3142a	Pr	3151	Ag	3159	Th	3167a	Y
3102a	Sb	3110	Ce	3118a	Gd	3127a	La	3135a	Nd	3143	Re	3152a	Na	3160a	Tm	3168a	Zn
3103a	As	3111a	Cs	3119a	Ga	3128	Pb	3136	Ni	3144	Rh	3153a	Sr	3161a	Sn	3169	Zr
3104a	Ba	3112a	Cr	3120a	Ge	3129a	Li	3137	Nb	3145a	Rb	3154	S	3162a	Ti		
3105a	Be	3113	Co	3121	Au	3130a	Lu	3138	Pd	3147a	Sm	3155	Ta	3163	W		
3106	Bi	3114	Cu	3122	Hf	3131a	Mg	3139a	P	3148a	Sc	3156	Te	3164	U		
3107	B	3115a	Dy	3123a	Ho	3132	Mn	3140	Pt	3149	Se	3157a	Tb	3165	V		
3108	Cd	3116a	Er	3124a	In	3133	Hg	3141a	K	3150	Si	3158	Tl	3166a	Yb		

Certified Concentrations:

Hg 9.96 ± 0.06 µg/ml

Note: The uncertainty of the certified value has been calculated from applicable uncertainty contributors (u_i) such as the SRM inherited uncertainty, weighing and dilution errors and instrument variability. The combined uncertainty ($u_c = \sqrt{2u_i^2}$) has been multiplied by a coverage factor (k) of 2 to provide a 95% confidence interval.

3.0 REFERENCE VALUES:

Density: **1.051 g/ml** @ **20.9 °C**

4.0 APPROVAL AND DATE OF CERTIFICATION:

Certification Approval: Yaling Sui, Chemist Yaling Sui
Certification Date: **April 19, 2013**

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C.O.C. CERTIFICATE OF ANALYSIS

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

ACCREDITED Testing Laboratory Certificate 863.01

ACCREDITED Reference Material Producer Certificate 863.02

2.0 **DESCRIPTION OF CRM** **10 µg/mL Mercury in 10% (v/v) HCL**

2035815
ID: ME_Hg_00019
Exp: 04/01/14 Prpd: CDC
Mercury Calibration Stand

Catalog Number:	MSHG-10PPM
Lot Number:	F2-HG02097
Starting Material:	Hg metal
Starting Material Purity (%):	100.0000
Starting Material Lot No:	R307HGA1
Matrix:	10% (v/v) HCL

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 9.990 ± 0.074 µg/mL

Certified Density: 1.026 g/mL (measured at 20 ± 1°C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

Certified Value $(\bar{x}) = \frac{\sum x_i}{n}$ ($\bar{x}$) = mean

x_i = individual results

n = number of measurements

Uncertainty (±) = $2\,[\,\Sigma(s_i)^2\,]^{1/2}$ 2 = the coverage factor.

$[\,\Sigma(s_i)^2\,]^{1/2}$ = The square root of the sum of the squares of the most common errors (where 's' stands for the standard deviation) from instrumental measurement, density, NIST SRM uncertainty, weighing, dilution to volume, homogeneity, long term stability and short term stability.

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed.,

Title: Purge and Trap for Aqueous Samples, SW846 Method 5030B and 5030C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Mary Baron Department Manager	2/9/21 Date	Dan Helfrich Health & Safety Manager / Coordinator	2/9/21 Date
Carl Armbruster Quality Assurance Manager	2/9/21 Date	Mark Acierno Laboratory Director	2/9/21 Date

Sylvanus Klusey — 2/9/21 Date
Organics Operations Manager

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method describes purge and trap techniques for the analysis of volatile organic compounds in aqueous samples by SW846 Method 5030B and 5030C and high concentration soil sample extracts prepared using SW846 Methods 5035 or 5035A.

1.1.2 SW846 Methods 5030B and 5030C can be used for most volatile compounds that have boiling points below 200° C and are insoluble or slightly soluble in water. Water-soluble compounds can be analyzed for by this method; however, quantitation limits will be higher due to poor purging efficiency.

1.1.3 This sample introduction step is to be followed by determinative methods such as SW-846 Method 8015 for GC analysis or SW-846 8260 for GC/MS analysis.

1.1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 9 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 An aliquot of the aqueous sample is purged with helium (or nitrogen, where applicable) as in a closed sparging vessel. For medium or high level samples, an aliquot of the extract prepared by SW846 Methods 5035 or 5035A is combined with organic free reagent water and then purged. The volatile compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is complete, the sorbent trap is heated and backflushed with helium (or nitrogen, where applicable) to desorb the volatiles onto a gas chromatographic column.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method and the determinative methods that follow are susceptible to contamination from a number of sources. Potential sources of contamination include organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum

during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatile laboratory is housed in a separate building, away from the organic extraction area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatile laboratory.

4.3 The helium (or nitrogen, where applicable) used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 A trip blank prepared from organic-free reagent water is carried through the sampling, storage, and analysis of each group of samples to check for such contamination.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Purge vessels are removed from the autosampler units after each use, rinsed, baked, returned to the units and pre-purged before the next use.

4.7 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.8 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

- Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

- Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

Purge and trap unit that consists of three parts: the sample purger, the trap, and the concentrator.

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required.

6.1.2 Purge and trap units used include the the OI 4451 autosampler/4560 concentrator, the Archon 5100A automatic sampler/OI 4560 concentrator, OI 4551A autosampler/4660 Concentrator, Centurion

Autosampler/Encon, Archon/Encon, Solatek autosampler/Stratum, Aquatek 70/Tekmar 3000 concentrator and Archon/Evolution.

6.1.3 The purge chambers of each unit are designed to accept a 5mL sample with a water column at least 3cm deep. The headspace above the water has a volume less than 15 mL. The purge gas is introduced no more than 5 mm from the base of the water column. The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3mm at the origin.

6.1.4 Different sorbent traps are used, depending on the determinative method that follows the purge and trap procedure. Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 The VOCARB 3000 (Supelco) is used in the Encon, Tekmar 3000 and Evolution. This trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001.

6.1.6 The OI Analytical purge trap #10 is used in the OI 4560 and 4660. This trap is 25cm long and has an inside diameter of 0.105 inches. The trap is packed to contain the following adsorbents: Tenax/silica gel/ carbon molecular sieve.

6.1.7 The concentrator of each unit is capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.8 Gas chromatograph. HP 5890/Agilent 6890N/7890 equipped with temperature programming capability.

6.1.9 GC column:

- 75M long x 0.53mm ID, J&W DB-624 capillary column with 3um film thickness or similar phase.
- 20M long x 0.18 ID, DB-624 or Rtx-VMS with 1 um film thickness.
- 30M long x 0.25 mm ID x 1.4 um film thickness Rtx-624.

6.1.10 Injection port liners. HP 18740-80200 or equivalent.

6.2 Supplies

- Microsyringes. 10 uL to 1000 uL.
- Syringes. 5mL and 30mL gas-tight.
- Volumetric flasks. Class “A” glassware, 10mL, 50mL, and 100 mL.

- Vials. 2mL amber glass with screw cap with Teflon-faced septa.
- VOA vials. 40ml glass with PTFE-faced septa

7.0 Reagents and Standards

7.1 Organic free reagent water. Distilled water purchased from Poland Spring.

7.2 Methanol: Purge and trap grade, purchased from JT Baker. (Cat # 9077-02)

7.3 Standard preparation and use is described in detail in each of the determinative methods.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Aqueous samples are collected in the appropriate containers and preserved according to the determinative method requirements.

8.2 All aqueous samples are protected from light and stored at 4°C in an area free of organic solvents. No standards or solvents are stored in the sample refrigerators or in the surrounding area.

8.3 All aqueous samples are analyzed within 14 days of collection.

8.4 The collection, preservation and hold times for soil samples prepared via SW846 Method 5035 or 5035A are addressed in the corresponding TestAmerica Edison SOPs (see references in Section 15).

9.0 Quality Control

9.1 Specific quality control procedures are outlined in each of the determinative methods that follow this purge and trap technique of sample introduction. Standard quality assurance practices are used with all methods.

9.2 An initial demonstration of accuracy and precision is performed for each determinative method. This demonstration is done for each sample introduction technique.

9.3 Reagent water blanks are analyzed to ensure that reagents and/or sample dilutions are free of interferences.

9.4 Method blanks are prepared and analyzed in the same manner as samples, and are carried through the entire analytical procedure to show that each system is in control.

9.5 Each analysis batch contains spike and duplicate data as outlined in the method, as well as a laboratory control sample (blank spike) to show that replicate data can be generated and appropriate concentrations of target analytes recovered.

10.0 Procedure

10.1 This section provides guidance for the analysis of aqueous samples and/or methanol extracts prepared by SW846 Method 5035 by purge and trap techniques. Determinative methods SW846 Methods 8015 or 8260 will follow this purge and trap procedure

10.2 Instrument operating parameters are set at the beginning of a method of analysis and remain constant throughout the calibration and sample analysis steps. See each determinative method to verify specific instrument operating parameters.

Table 1: Example Instrument Operating Parameters

Purge and trap unit	
Purge Time	11 minutes
Dry Purge	1 Minutes
Purge Gas	Helium (or nitrogen, where applicable)
Purge Flow	40-45 ml/min
Purge Temp	Water: Ambient, Solids: 40^{0}C
Trapping Temp	Ambient, <30^{0}C
Desorb Time	1 Minute
Desorb Temp	VOCARB: 260^{0}C
Gas chromatograph	
Injector	180^{0}C
Carrier Gas	Helium
Carrier Flow	6 ml/min
Oven Program	35 - 250^{0}C with 2 ramps
Run Time	26 Minutes
Mass Spectrometer	
Electron Energy	70 volts (nominal)
Mass range	35-260 AMU
Scan time	0.9 sec./scan
Source Temp	200^{0}C
Separator Temp	180^{0}C

10.3 Screen all samples according to TestAmerica Edison SOP No. ED-GCV-001, (*Screening for Volatile Organics, Static Headspace with GC FID,, SW846 Method 5021* **,** current revision). Analysts are furnished with approximate dilution factors prior to analysis.

10.4 Allow all samples and standards solutions to warm to ambient temperature before use.

10.5 Prior to performing any purge and trap analysis, prepare each system in the following manner:

10.5.1 Condition concentrators by baking the trap at their bake temperatures for 5-12 minutes. Also condition the GC column by baking at 200°C.

10.5.2 Access the FLUSH menu on the Archon Autosampler. Rinse the syringe barrel with two 6mL portions of rinse water. Turn off the helium switch, and fill the water reservoir with reagent water. Visually inspect the standard vial and fill with the appropriate internal standard/surrogate mix. Turn the helium switch back on. Prime the standard loop by accessing the SYSTEM/MAINTENANCE/STANDARD CONTROL menu. Empty the waste reservoir.

10.5.3 Prepare other referenced autosamplers by filling internal standard reservoirs with the appropriate IS/SURR, empty the waste reservoir and fill the water rinse reservoir.

10.6 Prior to using this sample introduction technique, the GC or GC/MS system must be tuned and calibrated as required by each of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15). All method requirements for tune, calibration, and blank analysis must be met prior to sample analysis.

10.7 Aqueous samples

10.7.1 Load the sample vials directly into the autosampler tray. The appropriate internal standard and/or surrogate solution will be added to the sample by the autosampler unit immediately before a sample is purged.

10.7.2 The process of using autosamplers also destroys the validity of the sample by puncturing the septum. Any re-analysis should be done using second vial of sample. If only one vial exists, do not use this autosampler.

10.8 Medium or high level sample

10.8.1 Prepare samples by adding the appropriate amount of sample to a 50mL volumetric flask containing reagent water.

10.8.2 Add the appropriate amount of sample to the flask. Dilute to volume with reagent water, cap, and invert three times. Pour the diluted sample into a 5mL syringe or a 40mL VOA vial.

10.8.3 If a dilution is required, reduce the volume of sample added to the

10.8.4 Load sample onto the autosampler.

10.8.5 Purge the sample for 11 minutes, with one minute dedicated to removing moisture from the purge and trap system (dry purge). Times may vary among different determinative methods.

10.8.6 After purging is complete, desorb the sample onto the GC column by rapidly heating the trap to 260°C and backflushing it with helium (or nitrogen, where applicable). (Trap temperatures will vary based on type used)

10.8.7 Begin the GC temperature program and data acquisition.

10.8.8 Re-condition the trap by baking at 260°C for 8-12 minutes.

10.8.9 Cool the trap to (<31°C). The trap is now ready for the next sample.

10.8.10 Process data using Chrom software, and evaluate as per each determinative method.

10.9 Technical acceptance criteria for sample analysis.

10.9.1 The samples must be analyzed on a GC or GC/MS system meeting the initial calibration, continuing calibration and blank technical acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15).

10.9.2 The sample must be analyzed within the required holding time.

10.9.3 The sample must have an associated method blank meeting the blank technical acceptance criteria.

10.9.4 The percent recovery of each of the system monitoring compounds in the sample must be within the acceptance windows.

10.9.5 After analyzing a sample that exceeds the initial calibration range the analyst must either analyze an instrument blank (using the same purge inlet if using an auto sampler) which must meet technical acceptance criteria for blank analysis or monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample that exceeded the calibration range.

10.10 Corrective Action for Sample Analysis

10.10.1 Samples must meet technical acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15) before reporting data.

10.10.2 Corrective action must be completed prior to sample analysis if analysis failed to meet instrument performance checks, initial calibration, continuing calibration and method blanks.

10.10.3 Corrective action for system monitoring compounds and internal standard compounds that fail to meet acceptance criteria must be completed prior to sample analysis.

10.11 If any of the system monitoring compounds and internal standard compounds fail to meet acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15):

10.11.1 Check all calculations, instrument logs, the system monitoring compound and internal standard compound spiking solutions and the instrument operation. If the calculations were incorrect, correct calculations and verify that the system monitoring compound recoveries and internal standard compound responses meet acceptance criteria

10.11.2 Check the preparation of the internal standards and system monitoring compounds for concentration and expiration.

10.11.3 Verify that the instrument is operating correctly.

10.12 Data that fails to meet minimum acceptance criteria will be annotated (flagged) with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a *Non-Conformance Memo* (NCM) (TestAmerica Edison Work Instruction No. EDS-WI-012) will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *Standard Operating Procedure for Control of Non-Conformances and Corrective Action* (TestAmerica Edison SOP No. EDS-GEN-003).

11.0 Calculations / Data Reduction

11.1 For specific calculations refer to the applicable determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0 Pollution Control

13.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2 The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0 Waste Management

14.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.2 The following waste streams are generated as a result of this analysis:

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system.

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene

waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium
  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

- Returned Methanol Preservative - Methanol preserved vials are collected in satellite accumulation and then transferred to 55 gallon open top steel waste drums in the waste room. These waste drums are then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, *Method 5030C: Purge and Trap for Aqueous Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3 2003.

15.2 United States Environmental Protection Agency , *Method 5035A*: *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 0, July 2002.

15.3 United States Environmental Protection Agency, “Method 8260D, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”, Test Methods for Evaluating Solid Waste, SW846, Update VI, Revision 4, June 2018.

15.4 United States Environmental Protection Agency, *Method 8015D*: *Non-Halogenated Organics using GC-FID,* Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Revision 4, June 2003.

15.5 TestAmerica Edison SOP No. ED-GCV-008, *Gasoline Range Organics using GC-FID Method, SW846 Method 801D,* current revision.

15.6 TestAmerica Edison SOP No. ED-MSV-002, *Closed System Purge and Trap and Extraction for Volatile Organics in Soil, SW846 Method 5035A,* current revision.

15.7 TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021* **,** current revision.

15.8 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.9 TestAmerica Edison SOP No. EDS-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action*, most current revision

16.0 Method Modifications

Not applicable

17.0 Attachments

Not applicable

18.0 Revision History

Revision 10, dated: 02/09/2021

- Updated to Eurofins branding;
- Updated Lab Quality Manual section references as applicable throughout
- Updated Section 15 (References) as needed.

Revision 9, dated: 06/26/2013:

- Revised title of SOP to include reference to SW846 5030C.
- Section 1.1.1 and throughout document as appropriate: added reference to SW846 5030C.
- Section 1.1.1: inserted word ‘extracts’ in reference to analysis of high concentration and soil samples by SW846 5035/5035A.
- Section 10.8.10: replaced Target reference with Chrom (newly implemented chromatography data system)
- Section 15.3: corrected method reference to ‘5035A’.
- Section 15.14: added reference to SW8465030C

Revision 8, dated: 11/27/2012:

- Updated referenced Lab Quality Manual section and appendix numbers as appropriate.

Revision 7, dated: 10/02/2008

- Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).

- Section 1.2 Added reference to Quality Assurance Manual for method modifications. Added references to related methods and SOPs for determinative methods.
- Section 2 (and throughout document): added option of purging with nitrogen gas as applicable.
- Section 3: revised to reference new location for definitions.
- Section 5: Revised to include most up to date corporate health and safety references and information.
- Section 6: Newer instrumentation and GC column types added. See sections 6.1.2 and 6.1.9. All references to Tekmar instruments 2016 and 2000 and procedures to use with it removed in all relevant sections.
- Section 11: Removed reference to Organic Calculation SOP.
- Section 12: updated and revised the MDL requirements to reflect text in the current revision of the TestAmerica Edison Laboratory Quality Manual (LQM).
- References: Expanded to include more specific SOP references
- Section 18: Added this Revision History section

Title: Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A

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1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits:** SW846 Method 8151A provides extraction and derivatization procedures for the gas chromatographic analysis of chlorinated acid herbicides in water, soil, and waste samples. An option for the hydrolysis of esters is also described. This SOP details the extraction of water samples with diethyl ether follower by esterification with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to TestAmerica Edison SOP No. ED-GCS-00, *Analysis of Organochlorine Herbicides by SW846 Method 8151A,* current revision.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

This SOP provides extraction and derivatization procedures for the determination of chlorinated acid herbicides and their esters in water and aqueous waste samples.

Water samples are hydrolyzed, cleaned with methylene chloride, acidified, extracted with diethyl ether and then esterified with Trimethylsilyl)diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD).

The sensitivity of Method 8151A depends on the level of interferences in addition to instrumental limitations.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing reagent blanks.

Matrix interference may be caused by contaminants that are co-extracted from the sample. Organic acids, especially chlorinated acids, cause the most direct

interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

The acid forms of the analytes are strong organic acids that react readily with alkaline substances and can be lost during sample preparation. Glassware and glass wool must be acid-rinsed with 1 N hydrochloric acid and the sodium sulfate must be acidified with sulfuric acid prior to use to avoid analyte losses due to adsorption.

Sample extracts must be dry prior to methylation or else poor recoveries will result.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Ethyl Ether is an extremely flammable solvent. If a mechanical device is used for sample extraction, the device should be equipped with an explosion proof motor and placed in a hood.

Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.

Diazomethane can explode under certain conditions therefore take the following precautions:

- Use a well-ventilated hood.
- Use a safety screen.
- Do not heat above 90°C.
- Avoid grinding surfaces, ground glass joints, sleeve bearings, and glass stoppers.
- Store away from alkali metals.
- Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

Ether creates excessive pressure very rapidly. Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm-TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. **May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of within six months.**

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Equipment

- Top loading Balance - Sargent Welch S2642-25 or equivalent
- Automatic separatory funnel rotator
- Steam Bath - Fisher Scientific 66738 or equivalent.
- Furnace - Thermolyne Type 6000 or equivalent.
- Vacuum Pump - General Electric Model No. 0322-V48-G180X or equivalent.
- N-Evap - Meyer Analytical Evaporator Model No. 112 or equivalent

6.2. Supplies

- 400 ml Clear Glass Jar
- 1 L Erlenmeyer Flask
- 500 ml Erlenmeyer Flask
- 2 L Separatory Funnel
- 500 ml Separatory Funnel
- 500 ml Centrifuge Bottle - Pyrex 1260 or equivalent.
- 10 ml graduated Concentrator Tube - Kontes K570050-1025 or equivalent.
- 10 ml glass vials with Teflon lined screw caps.
- 20 ml glass vials with Teflon lined screw caps.
- 5 ml to 1000 ml Class A Volumetric flasks
- Funnel - 75 mm diameter

- 3 Ball Macro Snyder Column - Baxter K503000-0121 or equivalent.
- 1 L Graduated Cylinder
- 50 ul Gastight Syringe - VWR 60376-241 or equivalent.
- 100 ul Gastight Syringe - Baxter S9662-5 or equivalent.
- 100 ml Graduated Cylinder
- 1 ml Pasteur Disposable Pipet
- Kuderna Danish (K-D) - Kontes 500 ml - Baxter K570001-0500 or equivalent.
- 10 ml Disposable Pipette
- Glass stopper - size 24/40
- Teflon stopper - size 19/22
- Filter paper - 15-cm diameter, Whatman No. 1 or equivalent.
- Phosphoric Acid Washed Glass Wool - Supelco 2-0383 or equivalent.
- Desiccator - Baxter D1420-2 or equivalent.
- Septa, 12 mm - Supelco 2-3273 or equivalent.
- Septa, 8 mm - Supelco 2-3272 or equivalent.
- Standard Taper Clamp (size 19, blue) - Wheaton 297749 or equivalent.
- Silicon Carbide Boiling Stones - Thomas 1590-D33 or equivalent.
- pH paper, Fisher 9590
- EM Quant Peroxide Test Strips, EMD No. 10011-1 or equivalent

Note: All glassware must be pre-rinsed with a 25% Sulfuric Acid: reagent water solution.

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

- Organic-free reagent water.
- Trimethylsilyldiazomethane (TMSD) , 2.0 molar solution in hexane, Acros 429211000 or equivalent
- Diethyl Ether (Residue Analysis Grade with 2% Ethanol preservative) - Baker 9259-03 or equivalent (tested for peroxide formation upon opening and monthly thereafter by use of EM Quant Peroxide Test Strips)
- Methylene Chloride (High Purity Grade) - Baker 9266 or equivalent.
- Acetone (High Purity Grade) – Baker 9254 or equivalent.
- Sodium Sulfate Anhydrous Crystals (Reagent Grade) – Malinckrodt 8024 or equivalent. (Baked at 400°C for 4 hours)
- Hexane (High Purity Grade) – Baker 9262 or equivalent.
- Carbitol (diethylene glycol monoethyl ether)
- Potassium Hydroxide (Analytical Reagent Grade) – Aldrich 306568 or equivalent
- Sulfuric Acid (Analytical Reagent Grade) - Mallinckrodt 2876 or equivalent.
- Silicic Acid, Aldrich 30, 636-3 or equivalent

7.1.1 Reagent preparation

7.1.1.1 Anhydrous sodium sulfate crystals must be baked in the muffle furnace at 400° C for 4 hours prior to use. After baking, the sodium sulfate must be acidified. Place 500 grams of anhydrous sodium sulfate into a 500 ml wide mouth Erlenmeyer flask. Add enough diethyl ether to cover the sodium sulfate. Add 0.5 ml concentrated sulfuric acid. Shake vigorously for 1 minute. Evaporate the ether by placing the flask on steam bath. Mix 1 g of the resulting solid with 5 ml of organic-free reagent water and measure the pH of the mixture. It must be below a pH of 4. Prepare fresh daily.

7.1.1.2 37% KOH Solution: Rinse a 1 liter Pyrex flask three times with organic free reagent water. Weigh 148 g KOH into flask and add 400 ml organic free reagent water. Mix until the KOH is completely dissolved and the flask has cooled to room temperature. Transfer the KOH solution to a clean 500-ml amber bottle and label.

7.1.1.3 12N H_2SO_4 : Rinse a 1 liter Pyrex flask three times with organic free reagent water. Add 200 ml organic free reagent water to the flask. Slowly, and while mixing, add 100-ml concentrated H_2SO_4 to flask. Continue mixing until flask has cooled to room temperature. Transfer the H_2SO_4 solution to a clean 500-ml amber bottle and label.

7.1.1.4 1:1 H_2SO_4 : Rinse a 1 liter Pyrex flask three times with organic free reagent water. Add 100 ml organic free reagent water to the flask. Slowly, and while mixing, add 100-ml concentrated H_2SO_4 to flask. Continue mixing until flask has cooled to room temperature. Transfer the H_2SO_4 solution to a clean 500-ml amber bottle and label.

7.1.1.5 25% H_2SO_4 solution (1:3 H_2SO_4 solution): 25 ml Acid and 75 ml reagent water. Add 75 ml organic free reagent water to a graduated cylinder. Slowly, and while mixing, add 25-ml concentrated H_2SO_4 to cylinder. Continue mixing until cylinder has cooled to room temperature. Transfer the H_2SO_4 solution to a clean amber bottle and label.

7.2 Standards

7.2.1 Standards are purchased as concentrated solutions (see Section 7.2.2). The surrogate stock solution is diluted (in volumetric glassware) to working concentration using acetone as the diluent as described in Section 7.3.

7.2.2 **Standard mixes and sources:** Table 1 lists the standard concentrate mix sources. Attachment 1 details the individual components of these mixes.

Table 1:
Herbicide Standard Mixes and Sources*

Standard Name ("Lab Name")	Concentration (ug/ml)	Source
Acid Herbicide LCS RTS ("Spiking Mix")	varies (in Methanol)	Restek Catalog # 567950
DCAA standard (Surrogate Spiking Solution)	1000 (in methanol)	Restek Catalog #567804

*May be substituted with equivalent standards from alternate sources.

Table 2:
Components of Herbicide Standard Mixes

Parameter	Catalog Nos.	Lab Standard Name	Concentration (ug/ml)
2,4-dichlorophenoxyacetic acid (2,4-D)	Restek Catalog # 567950	Spiking Mix	20
2-(2,4,5-Trichlorophenoxy)propionic acid (2,4,5-TP aka Silvex)	Restek Catalog # 567950	Spiking Mix	5
2,4,5-T	Restek Catalog # 567950	Spiking Mix	5
2,4-DB	Restek Catalog # 567950	Spiking Mix	20
Dalapon	Restek Catalog # 567950	Spiking Mix	20

Table 2: Components of Herbicide Standard Mixes			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Dicamba	Restek Catalog # 567950	Spiking Mix	10
Dichloroprop	Restek Catalog # 567950	Spiking Mix	20
Dinoseb	Restek Catalog # 567950	Spiking Mix	20
MCPA acid	Restek Catalog # 567950	Spiking Mix	2000
MCPP acid	Restek Catalog # 567950	Spiking Mix	2000
Pentachlorophenol	Restek Catalog # 567950	Spiking Mix	5
Picloram (4-amino-3,5,6-trichloropicolinic acid)	Restek Catalog # 567950	Spiking Mix	20
2,4-Dichlorophenylacetic Acid	Restek Catalog # 567804	Surrogate Spiking Solution	1000

7. 3. Standards Preparation

7.3.1. The surrogate stock solutions are diluted to the working concentrations with acetone as indicated in Attachment1 and 2.

7.3.2. Herbicide Spiking Standard (LCS/MS/MSD):

7.3.2.1 Herbicide QC Spiking Standard Mix (12 compound analysis): The 12 compound spiking standard is ready to spike and used when analyzing for the standard list of herbicides The spiking standard mix is a 25 ml solution (Restek Catalog # 567950; see Tables 1 and 2) of the 12 compounds at the specified concentrations as free acids. The concentrate is used without further dilution prior to the spiking of the following quality control sample types: MS, MSD, and LCS. Spiking instructions are found in Section 10.

7.3.3. Herbicide Surrogate Spiking Standard (2,4-DCAA Acid Spike Mix**):** The herbicide surrogate spiking standard is prepared as detailed in Attachment 1 using volumetric glassware and the concentrated free acid herbicide standards listed in Table 1 above.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass	1L amber/ 8oz amber	Cool 4 ± 2°C	7 days to extraction; 40 days to analysis	USEPA SW846

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2°C) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated

acceptance criteria. If the spiked sample recovery results fall outside the laboratory generated limits (refer to the current active TALS method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in TestAmerica Edison SOP No. ED-GCS-00, *Analysis of Organochlorine Herbicides by SW846 Method 8151A,* current revision.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing 2,4-DCAA (see Section 7.3.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as "estimated concentration".

10.0. Procedure

10.1. Sample Extraction (Aqueous Samples)

Note: For TCLP or SPLP leachate extraction procedure proceed to Section 10.6

10.1.1. Rinse enough 2-liter/500ml separatory funnels and 500-ml Erlenmeyer flasks to accommodate the number of samples to be prepped. Each funnel and flask must be rinsed four times: once with a 50:50 mixture of methylene chloride:acetone, twice with methylene chloride and once with a 25% sulfuric acid: reagent water solution.

10.1.2. Mark the lab sample identification numbers on the corresponding separatory funnels with red wax pencil.

10.1.3. Make up tags for each sample with the following information and place on the corresponding 500 ml Erlenmeyer flasks.

HERBICIDES
Sample Number
Matrix
Date of Extraction

10.1.4. Mark the fluid level of sample bottles (volume) with a black magic marker or red wax pencil. Pour each sample into its corresponding separatory funnel. Fill each sample bottle to the black line with tap water. Pour this into the graduated cylinder used for measuring sample volumes. Note the volume for each sample on the Organic Extraction Data Sheet or enter directly into the TALs batch record.

10.1.5. Rinse a 1000 ml graduated cylinder once with methylene chloride and two to three times with lab reagent water. Using the graduated cylinder obtain 1000 ml of lab reagent water from the Millipore filtering apparatus for each of the method blank and the laboratory control sample (LCS) (aka blank spike).

10.1.6. Pour the reagent water for the method blank and LCS into the corresponding, labeled separatory funnels.

10.1.7. Rinse each syringe to be used for adding surrogates and spikes, eight to ten times with acetone.

10.1.8. Add 50 ul of 100 ppm 2,4-Dichlorophenylacetic Acid (DCAA) Surrogate Spiking Solution (see Attachment 2) to each sample and QC sample. NOTE: when spiking the samples with either surrogates or target spiking compounds, ensure that there are no bubbles in the syringe. In addition, hold the syringe just above the level of the water when adding the spike. Do not touch the tip of the syringe to the water or to the side of the separatory funnel. Swirl the separatory funnel after adding surrogate spike. All spikes must be witnessed.

10.1.9. When preparing QC spike samples (MS/MSD/LCS), spike 500ul of 12 compound mix (see Tables 1 and 2).

10.1.10. Add 2 ml of 1:1 Sulfuric Acid/ reagent water to each sample and QC sample. Shake to mix. Check pH with pH paper. The pH must be ≤ 2. Add sulfuric acid as needed to obtain proper pH. Note: pH adjustments must be documented in the extraction log and/or in the TALS batch record.

10.1.11. Add 150 ml of diethyl ether to each sample bottle. Rinse the bottle thoroughly with the ether and transfer the ether to its corresponding separatory funnel.

10.1.12. Extract the samples by vigorously shaking the separatory funnels for 2 minutes, with periodic venting to release excess pressure.

10.1.13. Allow the organic layer to separate from the water phase for a minimum of 10 minutes.

10.1.14. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation or other physical methods.

10.1.15. Collect the diethyl ether layer for each sample into the corresponding 500-ml Erlenmeyer flask.

10.1.16. Repeat steps 10.11 through 10.15 twice, using 50-ml aliquots of diethyl ether each time.

10.1.17. Discard the aqueous phase.

10.2. Hydrolysis

10.2.1. To each 500-ml Erlenmeyer flask containing the 200-ml ether extract, add 2 ml of the 37% KOH solution, 15 ml of reagent water, and 4-5 boiling chips.

10.2.2. Attach a three-ball macro Snyder and place Erlenmeyer flask on steam bath (100ºC) for 1½ to 2 hours.

10.3. Partitioning

10.3.1. Remove the Erlenmeyer flask from the steam bath and allow it to cool for 5-10 minutes.

10.3.2. Pour the extract into a 500-ml separatory funnel pre-rinsed with diethyl ether.

10.3.3. Add 40 ml of diethyl ether to the separatory funnel and shake for two minutes.

10.3.4. Pour the dried diethyl ether extract through a funnel plugged with acid washed glass wool, collecting the extract in the K-D concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.3.5. Repeat twice using 20-ml aliquots of diethyl ether.

10.3.6. Discard the ether phase.

10.3.7. Adjust the pH of the extract remaining in the 500-ml separatory funnel to ≤ 2 by adding 2 ml of 1:3 Sulfuric Acid. Check pH with pH paper.

10.3.8. Add 40 ml of diethyl ether to the separatory funnel and shake for 2 minutes with periodic venting to release pressure.

10.3.9. Collect each diethyl ether phase into a single pre-cleaned screw top glass jars each containing approximately 50 g of acidified sodium sulfate crystals and mix well.

10.3.10. Repeat sections10.3.8 and 10.3.9 twice using 20-ml aliquots of diethyl ether.

10.3.11. Allow the diethyl ether extract to remain in contact with the sodium sulfate for approximately 2 hours.

10.3.12. Pour the dried diethyl ether extract through a funnel plugged with acid washed glass wool, collecting the extract in the K-D concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.3.13. Place the white identification tag on the K-D apparatus.

10.3.14. Rinse the Erlenmeyer flask and funnel with 20 to 30 ml of diethyl ether to complete the quantitative transfer.

10.4. Extract Concentration

10.4.1. Add one or two clean boiling stones to the K-D flask and attach a three-ball Snyder column.

10.4.2. Pre-wet the Snyder column by adding about 1 ml of diethyl ether to the top of the column.

10.4.3. Place the K-D apparatus on a hot water bath (15-20°C above the boiling point of the solvent; set at LOW which is 60ºC) such that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor.

10.4.4. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes.

10.4.5. Boil the sample in the K-D flask on the steam bath until the sample volume is approximately 1 ml.

10.4.6. Add 60 ml of hexane and concentrate further to a final volume of approximately 5 ml on a hot water bath (Set at MEDIUM which is approximately 78ºC).

10.4.7. Remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

10.4.8. Remove the Snyder column from the top of the K-D flask. Remove the blue taper clamp from the ground glass joint and dry

the exterior with a Kimwipe. Remove the K-D flask from the concentration tube.

10.4.9. Place the concentration tube onto the N-Evap apparatus. Maintain I.D. tag with concentration

10.4.10. Continue to concentrate under a N2 stream until the sample volume is approximately 1 ml.

10.4.11. To each sample add 500ul of methanol and dilute to a final volume of 10 ml with hexane when the initial volume is 1L , to 3ml final volume when the initial volume is 250ml, and 5 ml final volume for TCLP extracts.

10.5. Esterification

10.5.1. Add approximately 200 uL of methanol to the extract followed by approximately 200 uL of 2.0M Trimethylsilyldiazomethane (TMSD) solution. The extract will turn a yellow color. If this does not occur, add additional (approximately) 200 uL aliquots of 2.0M Trimethylsilyldiazomethane (TMSD) solution until the yellow color persists. Check the sample every 15 minutes for yellow color. If the yellow disappears in this time frame, continue adding an additional aliquot of 2.0M Trimethylsilyldiazomethane (TMSD) solution until the yellow color persists.

10.5.2. Allow the extract to stand for at least one hour at room temperature to allow the methylation reaction to occur. The reaction is halted with the addition of approximately 0.05g (one scoop using a disposable spatula) of silica gel. The extract is then brought up to a final volume of 10 mL with hexane by visually comparing it to a calibrated collection tube.

10.5.3. Adjust the final extract volumes to 10ml with hexane. The extracts ready to be analyzed by GC. Transfer some volume to GC autosampler vials.

10.5.4. Samples must be analyzed as soon as possible (less than 24 hrs. preferably) after esterification. This is to minimize the trans-esterification and other potential reactions that may occur.

10.5.5. Store refrigerated if further processing will not be performed immediately. Extracts should be stored at 4°C away from light.

10.6. Sample Extraction (Leachate Samples)

10.6.1. Rinse enough 60-ml separatory funnels and 500-ml Erlenmeyer flasks to accommodate the number of samples to be prepped. Each funnel and flask must be rinsed four times: once with a 50:50 mixture of methylene chloride:acetone, twice with

methylene chloride and once with a 25% sulfuric acid: reagent water solution.

10.6.2. Mark the lab sample identification numbers on the corresponding separatory funnels with red wax pencil.

10.6.3. Make up tags for each sample with the following information and place on the corresponding 500 ml Erlenmeyer flasks.

HERBICIDES
Sample Number
Matrix
Date of Extraction

10.6.4. Pipette 15 ml of each sample into its corresponding separatory funnel.

10.6.5. Rinse a 100 ml graduated cylinder once with methylene chloride and two to three times with lab reagent water. Using the graduated cylinder obtain 15 ml of lab reagent water from the Millipore filtering apparatus for each of the method blank and the laboratory control sample (LCS) (aka blank spike).

10.6.6. Rinse each syringe to be used for adding surrogates and spikes, eight to ten times with acetone.

10.6.7. Add 50 ul of 100 ppm 2,4-Dichlorophenylacetic Acid (DCAA) Surrogate Spiking Solution (see Attachment 1) to each sample and QC sample. NOTE: when spiking the samples with either surrogates or target spiking compounds, ensure that there are no bubbles in the syringe. In addition, hold the syringe just above the level of the water when adding the spike. Do not touch the tip of the syringe to the water or to the side of the separatory funnel. Swirl the separatory funnel after adding surrogate spike. All spikes must be witnessed.

10.6.8. When preparing QC spike samples (MS/MSD/LCS), spike 500uL of 12 compound mix (see Tables1 and 2). .Swirl the separatory funnel after adding the spike solution(s).

10.6.9. Add 2 ml of 37% KOH solution to each sample.

10.6.10. Add 20 ml of diethyl ether.

10.6.11. Extract the samples by vigorously shaking the separatory funnels for 2 minutes, with periodic venting to release excess pressure.

10.6.12. Allow the organic layer to separate from the water phase for a minimum of 10 minutes.

10.6.13. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation or other physical methods.

10.6.14. Collect the diethyl ether layer for each sample into the corresponding 250-ml Erlenmeyer flask.

10.6.15. Repeat steps 10.6.10 through 10.14 twice, using 10-ml aliquots of diethyl ether each time.

10.6.16. Discard the ether phase after each extraction.

10.6.17. Adjust the pH to $\leq$ 2 by adding approximately 2 ml of 1:3 Sulfuric Acid/Water to the samples. Check pH with pH paper.

10.6.18. Extract three times for two minutes each with ether, using 20 ml the first time, and 10 ml each the second and third times.

10.6.19. Collect all three ether phases into a screw top 125-ml glass jar containing acidified sodium sulfate. Let the sample dry for 2 hours.

10.6.20. Pour the dried extract through a funnel plugged with acid washed glass wool, and collect the extract into the K-D flask. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.6.21. Place the white I.D. tag on the K-D apparatus.

10.6.22. Rinse the Erlenmeyer flask and funnel with 20 to 30 ml of diethyl ether to complete the quantitative transfer.

10.6.23. Proceed to Section 10.4.1.

10.7. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.7.1. The TALS reagent database must be completed in full with the required information whenever standards are logged and/or prepared.

10.7.2. All required data types (sample volumes, reagent and standard volumes, reagent and standard lots, etc…) must be entered into the TALS batch database each time an extraction is performed.

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11.0. Calculations / Data Reduction

None

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the TestAmerica corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months..

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to the current revision of TestAmerica Edison SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section

13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method 8151A: Organochlorine Herbicides by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. TestAmerica Edison SOP No. ED- ORP-023, *Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A,* current revision.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.4. TestAmerica Corporate Document No. CW-E-M-001, *Environmental Health and Safety Manual,* current revision.

15.5. TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.6. TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.7. TestAmerica Edison SOP No. ED-GEN-013, *Glassware Cleaning*, most current revision

15.8. TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision.

16.0. Method Modifications:

NONE

17.0. Attachments

Attachment 1 – Surrogate Solution Prep

18.0. Revision History

Revision 15, 10/5/2021

- Updated Lab Quality Manual section references as needed throughout document.
- Updated throughout to reflect used of TMSD for esterification (replacing diazald)
- Section 12: updated to reflect current MDL procedures.

Revision 14, April 26, 2019

- Section 10.4.11: methanol was added to extract to help with better herbicide recoveries.

- Revision 13, dated February 22, 2016:
  - Updated Table 2 with the corrected spelling of Picloram(4-amino-3,5,6-trichloropicolinic acid.

- Section 7.1.1.3: corrected the preparation instructions for 12N sulfuric acid. The correct volume of water is 200ml (was 300 ml). 100ml concentrated sulfuric diluted into 200 ml water is 12N.
- Section 7.1.1.5: clarified to indicate that a 25% H2SO4 solution is a 1:3 solution.

- Revision 12, dated January 28, 2016:

  - Sections 7.2.1 and 7.3.1: updated info to reflect prep using new standard solutions.
  - Tables 1 and 2 updated with new standard mixes and sources.
  - Attachment 1: deleted. Attachment 2 now becomes Attachment 1.
  - Section 7.3.2.1: revised to reflect new standards and procedures.
  - Section 7.3.3: updated to revise from Attachment 2 to Attachment 1.
  - Section 8.0: added 8 oz amber glass container as option.
  - Section 10.1.1: added 500 ml sep funnel as option.
  - Section 10.1.9 and 10.6.8: updated spiking instructions.
  - Section 10.4.11: updated and clarified final extract volumes.

- Revision 11, dated January 27, 2014::

  - Minor editorial changes throughout document.
  - Section 7.2.2: Update standard sources and mixes (Table 1).
  - Section 7.3.2 and 7.3.3: Updated standards prep information.
  - Section 10.1.9: updated QC spiking procedures (LCS/MS/MSD).
  - Section 10.6.8: updated leachate QC spiking procedures.
  - Added Attachment 1 which details the individual target compounds and their concentrations in the new standards.
  - Added Attachment 2 which details the individual surrogate compounds and their concentrations in the new standards.

- Revision 10, dated January 6, 2012:

  - Section 7.2.2, Table 2: updated concentration of MCPP spiking mix.
  - Section 7.2.2, Tables 1 and 2: Updated source of Picloram spiking standard.
  - Section 7.3.2.4: revised instructions for preparation of Picloram spiking solution.
  - Corrected number of Sections 7.3.2.1 thru 7.3.2.4.
  - Revised Section 10.6.8: corrected typos and added detailed procedures dependent upon spiking scenarios.

- Revision 9, dated December 21 2011:

  - Revised the following sections to reflect the requirement that all glassware must be rinsed with sulfuric acid prior to use in the extraction of herbicides: Section 6.2, Section 10.1.1 and Section 10.6.1.
  - Added Section 7.1.1.5 which describes the preparation of a 25% sulfuric acid: reagent water solution for use in acid-rinsing glassware.

- Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision

- Revision 8, dated August 26 2011:

  - Section 5.1: revised to include following statement: “Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.”
  - Section 6.2: added EM Quant Peroxide Test Strips to supplies list.
  - Section 7.1: added requirement to test Ether for peroxide formation.
  - Revised Section 10.3.4 to include a requirement to pour extract through a funnel plugged with acid washed glass wool. Also revised collection container (was Erlenmeyer flask; now K-D concentrator).
  - Section 10.3.9 was revised to clarify that the ether extracts from each sample are collected into a single container (not multiple separate containers).
  - Section 10.6.8: corrected the spike volume used for the 10 Compound Spike mix. Reworded the section to make the instructions clearer.

- Revision 7, dated October 30, 2009
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
  - Section 1.1: Expanded to include references to applicable prep and cleanup SOPs.
  - Section 3: revised to reference new location for definitions.
  - Section 5: Revised to include most up to date corporate health and safety references and information.
  - Section 7.2.2: Added tables detailing components found in the various standards mixes.
  - Section 7.3: Updated the instructions for preparation of standards.
  - Section 7.3: Added tables with calibration standards prep details.
  - Section 8: Updated with additional details including a table outlining containers, preservation and holding times for waters and soils.
  - Section 9.1: Expanded QC sample preparation details.
  - References: Expanded to include more specific SOP references
  - Section 18: Added this Revision History section
  - Throughout document: added references to TestAmerica LIMS (TALS).

Attachment 1 – Surrogate Standards Preparation

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Reagent ID: OP_HERB_SU_00020

		Expiration Date:	06/10/2014
Description:	HERBICIDE Surrogate	Laboratory:	TestAmerica Edison
No. of Bottles:	10	Prepared By:	Patel, Hemex
Storage Location:	ORP-STDS & SPIKES	Solvent:	Acetone
Reagent Volume:	100.000 mL	Solvent Lot:	33214
Creation Date:	12/10/2013		
Container(s):	2644797, 2644798, 2644799, 2644800, 2644801, 2644802, 2644803, 2644804, 2644805, 2644806		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
2,4-Dichlorophenylacetic acid	OP_2,4DCAA_00016	09/30/2014	1000.00000	ug/mL	100.00000	ug/mL

Source Reagents

Reagent	Description	Type	Expiration	Vendor	Vendor Lot #	Vendor Cat Lot #	Volume Used	Volume Units
OP_2,4DCAA_00016	2,4 - DCAA Acid Spike Mix	ASTD	09/30/14	RESTEK	A093820	567804	10.00000	mL

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Title: Digestion of Water and Wastewater Samples for Analysis by ICP and ICP-MS, SW846 Method 3010A

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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

Method SW846 3010A covers the preparation procedures for the determination of elemental constituents in water samples, wastewater samples and leachates by inductively coupled plasma (SW846 Method 6010D) and inductively coupled plasma – mass spectrometry (SW846 Method 6020B).

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

The elements analyzed for by this method and the laboratory's reporting limits (RLs) are summarized below:

Element	Method 6010D Water (ug/L)	Method 6020B Water (ug/L)
Aluminum	200	40
Barium	200	4
Beryllium	2	0.8
Boron	50	80
Cadmium	4	2
Calcium	5000	500
Chromium	10	4
Cobalt	50	4
Copper	25	4
Iron	150	120
Manganese	15	8
Magnesium	5000	200
Molybdenum	20	4
Nickel	40	4
Potassium	5000	200
Silver	10	2
Sodium	5000	500
Strontium	20	4
Tin	50	16
Titanium	20	4
Vanadium	50	4
Zinc	30	16
Antimony	20	2
Arsenic	15	2

Element	Method 6010D Water (ug/L)	Method 6020B Water (ug/L)
Lead	10	1.2
Selenium	20	2.5
Thallium	20	0.8
Silicon	500	n/a
Sulfur	200	n/a

2.0 Summary of Method

A volume of sample is refluxed with nitric acid until the digestate is light in color in a sample cup using a hot-block. After the digestate is reduced to a low volume, it is refluxed with hydrochloric acid and brought to a final volume for inductively coupled plasma (ICP) by SW846 Method 6010D or inductively coupled plasma-mass spectrometry (ICP-MS) analysis by SW846 Method 6020B.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Whenever reflux is required or sample is to be reduced, NEVER BOIL THE SAMPLE. Boiling could result in a significant loss of sample constituents.

4.2 See the current analytical SOPs for further information related to interferences: Eurofins SOP Nos. ED-MT-004 *Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy by using SW846 Method 6010D* and ED-MT-034, *SW-846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS*.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Block Digestor: Adjustable and capable of maintaining a temperature of 90°C-95°C (Environmental Express or equivalent).

6.2. Supplies

- watch glasses
- 75 mm funnels
- 50 ml and 100 ml Hot Block Digestion Cups
- Whatman # 41 filter paper or equivalent
- 0.45 micron filter units and vacuum pump
- Pipettes varying volumes: Eppendorfs & Fisher

7.0 Reagents and Standards

7.1. Reagents

7.1.1. Nitric Acid, Concentrated - Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.2. Hydrochloric Acid, Concentrated - Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.3. Reagent Grade Water 18 Megohm Minimum

7.2. Standards

7.2.1 Stock ICP Spike Standards:

7.2.1.1 Solutions A, B, C, and D (ICP-Spk), Part No. 4400-100419DD01: commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 1 for the standards certified concentrations and catalog numbers. 1000ppm each of Silicon and Sulfur purchased from CPI International or equivalent.

7.2.1.2 Solutions A, B, C, and D (TCLP-Spk), Part No. SM-606-114: commercially purchased from High-Purity Standards. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 1 for the standards certified concentrations and catalog numbers.

7.2.2 Stock ICP-MS Spike Standards:

7.2.2.1 ICPMS LCS/SPK, Part No. SM-606-111: commercially purchased from High-Purity Standards. Store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard doesn't require any pre-dilutions and is ready to be spiked

directly into the QC samples. See Attachment 1 for the standards certified concentrations and catalog numbers.

7.3 Working Spike Standards:

7.3.1. ME_LCS-int (for ICP prep) - Add the following to a 1000 ml volumetric flask and bring to volume using 5% HNO_3: 50 ml each of Part No. 4400-100419DD01 Solutions A, B, C, and D (Sec 7.2.1.1). Record standard preparations in TALS Reagent module. **Note**: Standard must not exceed the earliest expiration date of any one of its components.

7.3.2. ME_TCLPspk (for TCLP prep) – Add the following to a 200 mL volumetric flask and bring to a volume using 5% HNO_3: 40 ml each of Part No. SM-606-114 (TCLP-Spk) Solutions: A, B, C, and D (Sec 7.2.1.2). Record standard preparations in TALS Reagent module. **Note**: Standard must not exceed the earliest expiration date of any one of its components.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample[4] Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	Polyethylene, glass	50 ml	HNO_3 to pH < 2 prior to shipment; if not, acidify upon receipt in lab [2,3]	180 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.
[2] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥ 2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).
[3] Aqueous samples may be stored at room temperature.
[4] All containers must be pre-washed with detergents, acids and water.

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with every 20 samples or every batch of samples digested whichever comes first.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	See the Quality Control Section of the referring analytical SOP (i.e., SOP No. ED-MT-004 and ED-MT-034)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	
Matrix Spike (MS)[1]	1 in 20 or fewer samples	
Sample Duplicate (DUP)[1]	1 in 20 or fewer samples	
Matrix Spike Duplicate (MSD)	When requested by the client	

[1] The sample selection for MS and Sample Duplicate is random unless specifically requested by a client. Quality control samples prepared with each set of samples digested include Matrix Spike, Sample Duplicate, Method Blank, Laboratory Control Sample and Matrix Spike Duplicate (MSD: when requested by the client). Use the same environmental sample for the matrix spike and duplicate sample whenever possible. If insufficient sample volume is available, another environmental sample may be used for the duplicate sample.

9.2 Instrument QC

None

10.0 Procedure

10.1. Sample Preparation

10.1.1. Filtration Procedure for Dissolved Metals not filtered in the Field

10.1.1.1. The unpreserved sample must be filtered through a 0.45um filter unit as soon as practical after collection.

10.1.1.2. Collect the required volume of filtrate by using a 0.45um filter unit and a vacuum pump.

10.1.1.3. Acidify the filtrate with 1:1 HNO_3 to a pH of <2.

10.1.1.4. The method blank (MB) must be filtered and digested under the same conditions as the lab filtered samples.

10.1.2. During digestion, at least 2-3 ml of sample solution must be maintained in the digestion cup. Sample must never be allowed to go to dryness. If

sample should go to dryness, discard sample and redigest.

10.1.3. Pour out 50 ml of well-mixed sample into a 50 ml digestion cups *[note:100 mL hot block digestion cups can be used, except use the same sample volume (50 mL) for the entire preparation batch].* Label the cup with the sample number using a permanent marker. Prepare the QC samples as follows.

10.1.3.1. Method Blank (MB): Pour out 50 ml of deionized reagent water into the 50 ml digestion cup. Label the cup MB mm/dd/yy (this being the date the sample was prepared) and batch number. The Method Blank is carried through the complete digestion process.

10.1.3.2. Matrix Duplicate (DU): Label the digestion cup assigned for the duplicate sample with sample number and suffix 'DU'. Pour 50 ml of the well-mixed sample.

10.1.3.3. Matrix Spike Sample (MS): Measure 50 ml of well-mixed sample into the appropriately labeled 50 ml digestion cups (cup is labeled with the sample number and suffix 'MS'). The resulting elemental concentrations are listed in Attachments 2 & 3.

- For ICP preps (non-TCLP): spike the ICP Matrix Spike sample with 1 ml of ME_LCS-int (Sec 7.3.1). If Silicon and Sulfur are needed, spike 0.2 mL each of 1000ppm Sulfur and Silicon.
- For ICP and ICPMS preps (TCLP): spike the Matrix sample with 0.5 ml ME_TCLPspk (Sec 7.3.2). If Silicon and Sulfur are needed, spike 0.2 mL each of 1000ppm Sulfur and Silicon.
- For ICP-MS preps (non-TCLP): spike the sample with 0.25 ml ICPMS LCS/SPK (Sec 7.2.2.1).

10.1.3.4. Laboratory Control Sample (LCS): Pour out 50 ml of deionized reagent water sample into the appropriately labeled 50 ml digestion cups. Label the cup 'LCS' and the batch number. Spike the ICP-LCS and ICPMS- LCS (as appropriate) in the same way as the Matrix Spike Sample, see Sec. 10.1.3.3. Refer to Attachments 2 & 3 for final elemental concentrations.

10.1.4. Add 1.5 ml concentrated HNO_3, cover with ribbed watch glass, place digestion cup on hot block and reduce volume to approximately 5 ml. Do not allow sample to boil or dry.

10.1.5. Cool digestion cup and add another 1.5 ml concentrated HNO_3, cover cup with a watch glass, and return to hot block allowing a gentle reflux action.

10.1.6. Continue heating, adding additional HNO_3 until the digestate is light in color or does not change in appearance. Additional acid must not exceed to more than 1.5 ml. Allow the sample to cool down

10.1.7. Add 5 ml of 1:1 HCl and warm cup gently for 15 minutes to dissolve any precipitate or residue. Allow sample to cool down.

10.1.8. Wash down watchglass and digestion cup walls with deionized water. If necessary, accurately label a new 50 ml sample cup and filter samples through a #41 Whatman filter paper to remove any silicates and other insoluble material that could clog the nebulizer on the instrument.

10.1.9. Bring the sample volume up to 50 ml.

10.1.10. Enter all batch/sample information into TALS (see Sec 11.4. for instructions). Print the sample labels from the batch and attach to the corresponding sample cups. These labels contain information such as: Job & sample number, Batch number and container number.

10.2. Calibration

10.2.1. The volume of the 50ml and 100 ml hotblock digestion cups are verified for each lot received. The 50 ml and 100 ml volume verification with the appropriate Lot number is documented in the 'Metals Labware Volume Verification Logbook.'

10.3. Sample Analysis

10.3.1. Refer to the Analytical SOP, Eurofins Edison SOP No. ED-MT-004, and SOP No. ED-MT-034.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{} \times 100$$

[(orig. sample value + dup. sample value)/2]

11.3. Concentration:

Refer to Eurofins SOP No. ED-MT-004 and ED-MT-034.

11.4. Documenting and Reporting

11.4.1. Sample preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS).

11.4.1.1. Double click the TALS icon on the computer desktop. Enter your username and password and select Login.

11.4.1.2. Under TALS Menu, click the 'plus' sign next to Analyst. Double click Analyst Desktop II. Click the 'plus' sign next to Methods.

11.4.1.3. Right click the prep method that you are using to prep the samples. Select Create New Batch-From Scratch.

11.4.1.4. The analyst must enter the following information: Sample names (use scanners), initial and final sample volume, spike name and amount used, and all reagents and their corresponding lot numbers.

11.4.1.5. After saving the batch information in TALS, select Print-Batch Sheets (which must be submitted to metals analytical dept. along with the samples) and select Print-Batch Labels (which must be attached to the corresponding final digestate sample cups in the batch).

11.4.2. Record the following reagents and the volume used for sample preparation in the batch information page under "batch comments" in TALS Analyst Desktop II: concentrated nitric acid and 1:1 hydrochloric acid. Record reagents in TALS by opening the prep batch, click on "edit" and then right click to choose "view batch information." Enter the information in the "batch notes and batch comments" section.

11.4.3. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable preparation batch.

11.4.3.1. Open the preparation batch and click on the Edit tab above to enter the Edit Mode.

11.4.3.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.4.3.3. Acknowledge by filling in responses to all unacknowledged findings.

11.4.3.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.4.3.3.2. Fill in the appropriate comments in the response box, then hit 'OK.'

11.4.3.3.3. Acknowledge all finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization. The neutralization system consists of a 55-gallon poly open top container. There is also a local snorkel exhaust for venting any noxious odors. Once the liquid is transferred into the container, the contents are sparged with air and a representative pH is measured and the amount of material is recorded in the discharge log. The pH value is recorded in the discharge log, and the liquid is neutralized to a pH of 6-9 SU using food grade sodium bicarbonate. Once the secondary pH has been taken, confirmed and recorded, the discharge valve is opened to the municipal sewer system and the neutralization process is complete. The date, type of waste stream, and volume discharged are recorded on a discharge log.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  TSDF under Veolia Profile Number: 590598

- Soil Retain Samples - These containers are stored in the main sample-receiving laboratory cooler until they are designated for disposal. Once designated for disposal, the samples are transferred to the waste disposal area and placed into cubic-yard disposal containers, e.g., Jupiter-boxes. This waste is shipped directly to the TSDF under Veolia Profile Number 402535 and is subject to incineration.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; SW-846, Method 3010A.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision

15.4. TestAmerica Edison SOP No. ED-MT-004, *Trace Metals Analysis by Inductively*

Coupled Plasma Emission Spectroscopy by using SW846 Method 6010B, 6010C & 6010D; most current revision.

15.5. TestAmerica Edison SOP No. ED-MT-034, *SW-846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS,* most current revision.

15.6. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.7. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Certificate of Analysis of ICP and ICPMS stock standard.
Attachment 2: ICP: Laboratory Control Sample and Matrix Spike Final Concentration in Soln (ug/L), for Non-TCLP and for TCLP
Attachment 3: ICP-MS Matrix Spiking Solution, ICPMS LCS/SPK

18.0. Revision History

- Revision 13, dated 21 April 2023
  - Updated to Eurofins branding throughout the SOP.
  - Sec 1.1, Table: Updated the Reporting Limits for Ca and Na to reflect current lab practices
  - Sec 14.2: Updated waste management procedures to reflect current lab practices.
  - Sec 11.4.2: Added requirements to record all reagents used in sample preparation.

- Revision 12, dated 07 October 2020
  - Updated SOP header with Eurofins emblem.
  - Sec 3: Updated location of 'Definitions' to Appendix 5 of Lab Quality Manual.
  - Throughout the document removed SOP references to the analytical methods 6010B, 6010C, 6020, and 6020A.
  - Sec 1.1: Updated the Reporting Limits table to reflect current lab practices.
  - Sec 7.2.1.1: Added Si and S to the list of stock ICP spike standards.
  - Sec 10.1.3.3: Added MS spiking instructions for Si and S.
  - Sec 11.3: Removed reference to SOP ED-MT-033.
  - Sec 15.9: Removed reference WI# EDS-WI-007, Metals preparation data review checklist, not applicable.
  - Attachment 2: Added LCS and MS final concentration in solution for Si and S.

- Revision 11, dated 06 September 2018
  - Throughout the SOP - revised to reference the analytical method 6010D and 6020B.
  - Sec 11.4.2: Replaced data review instructions (Metals Data Review Checklist-EDS-WI-007) with TALS Data Review Checker (DRC); instructions added in SOP.

- Revision 10, dated 03 August 2016
  - Updated this SOP to reflect the new initial and final sample volumes (50ml) keeping the sample/reagent volume ratio the same; 100 ml initial and final sample volumes have been removed from this SOP.
  - Removed all reference to SOP ED-MT-033 from this SOP.
  - Sec 6.2: Added 50ml hotblock digestion cups to the list of supplies.
  - Sec 7.2.2.1: Added the part number for the ICPMS stock spike standard.
  - Sec 8.0: Revised the minimum sample size to 50ml.
  - Sec 9.1: Added matrix spike duplicate to the list of QC sample.
  - Sec 10.1.3.3: Revised the spiking amount (ml) added to the Matrix spike sample to reflect the 50 ml final volume.
  - Sec 10.14, Sec 10.1.5, Sec 10.1.6, and Sec 10.1.7 Updated the amount (ml) of reagents added to reflect the 50 ml final volume.
  - Sec 10.2.1: Added 50ml hotblock digestion cups.

- Revision 9, dated 19 August 2013
  - Sec 1 & 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 11.4.1.6. Added requirements to complete the Batch preparation section of the Metals Data Review Checklist (EDS-WI-007) prior to data submission.

- Revision 8, dated 24 August 2011
  - Sec 10.1.1.4: Added new section to include procedure for filtering method blanks.

- Revision 7, dated 13 April 2011
  - Sec 1.1: Revised to reference the analytical methods 6010C and 6020A and added their corresponding reporting limits to the Analytical Method Reporting Limits Table.
  - Sec 2.0, 10.3 & 11.3: Revised to reference the analytical methods 6010C and 6020A.
  - Sec 3: Revised LQM reference to the list of definitions.
  - Sec 4.2: Added SOP No. ED-MT-033 and ED-MT-034 for reference.
  - Sec 7.2.1 & 7.3: Added new spike standards for ICP (p/n 4400-100419DD01) and TCLP prep (TCLP-Spk).
  - Sec 8: Added Footnotes.
  - Sec 10.1.3.3: Added TCLP spiking instructions.
  - Sec 11.4: updated documenting and reporting instructions to comply with TALS procedure.
  - Sec 15: added applicable references.

- Sec 17: Attachment 2: Updated Table to include TCLP spike concentration; deleted internal COC (previously attached as Attachment 4).
- Updated COAs in attachment 1.

- Revision 6, dated 24 November 2008
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec 1.1: Deleted '*flame atomic absorption spectroscopy*.'
  - Added information related to ICP-MS (method 6020) where applicable (i.e. Sec 1.1, Sec 7 etc.).
  - Added Laboratory's reporting limits in Table format.
  - Sec 6: Updated list of equipment and supplies to include current materials used (e.g. added block digestor, deleted hot plate & centrifuge tubes).
  - Section 7: Included storage and stability information of reagents and standards. Updated the source of the stock spike standards and the preparation instructions for the ICP working spike standards.
  - Section 8: Updated in Table format and have included sample container, sample size requirements and method reference.
  - Sec 10: Deleted procedure for acid rinsing glassware; replaced use of beakers with disposable digestion cups
  - Deleted the use of 100 ml digestion cup and replaced with 125ml specimen cup.
  - Deleted procedure for transferring digestates from digestion cup to specimen cup. Also added information and procedure on internal chain of custody to identify sample digestates.
  - Section 15: Added applicable references
  - Section 17: Added attachment 1 to include certificate of analysis of stock standards, attachment 3 to include on instrument elemental concentrations of the ICPMS QC samples and attachment 4 to describe the internal chain of custody used for the digested samples.

Attachment 1

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SOLN. A

Expiry: 21-Sep-12

DE
4-4-11

Certificate of Analysis

Date Recd
03/22/11 MP

Part Number: 4400-100419DD01
Lot Number: 11C157
Shelf Life: 18 Months

TestAmerica/Edison
Custom Standard
5% HNO3

Concentrations in ug/mL ± 0.5%

Fe	1000	Zn	500
Se	2000	Mn	500
TL	2000	Ni	500
Be	50		
Cd	50		
Cr	200		
Co	500		
Cu	250		
Pb	500		
Ag	50		
Sr	500		
V	500		

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution B**
Lot Number: **11C157**
Shelf Life: **18 Months**

TestAmerica/Edison
Custom Standard
10% HNO3

Date Rec'd
03/22/11 MP

Concentrations in ug/mL ± 0.5%

Al	2000
Mg	20000
K	20000
Ca	20000
Na	20000

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution D**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
09/22/11 MP

TestAmerica/Edison
Custom Standard
2% HNO3

Concentrations in ug/mL ± 0.5%

As	2000
Ba	2000
B	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution C**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
03/22/11 MP

TestAmerica/Edison
Custom Standard
5% HNO3 + 2% HF

Concentrations in ug/mL ± 0.5%

Sb	500
Mo	500
Sn	500
Ti	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megachm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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Certificate of Analysis

SM-606-111
(ICPMS LCS/SPK)

Lot # 1031503

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts and Oxides	99.98+%	HNO_3, 4% + Tr HF	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp Date: NOV 15 2011
MSDS ATTACHED

SM-606-111
(ICPMS LCS/SPK)
Element List
(µg/mL)

Aluminum	500
Antimony	5
Arsenic	10
Barium	10
Beryllium	5
Boron	100
Cadmium	5
Calcium	500
Chromium	10
Cobalt	5
Copper	10
Iron	500
Lead	5
Magnesium	500
Manganese	50
Molybdenum	10
Nickel	10
Potassium	500
Selenium	10
Silver	5
Sodium	500
Strontium	10
Thallium	4
Tin	10
Titanium	10
Vanadium	10
Zinc	50

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Certificate of Analysis

Rec'd 4-11-11 DE

SM-606-114 (TCLP-Spk)
Solution A

Lot # 1109507

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts and Oxides	99.995+%	HNO_3, 4%	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

APR 0 7 2012

Theodore C. Rains, Ph.D.

SM-606-114 (TCLP-Spk)

Solution A

Element List

(µg/mL)

Barium	5000
Beryllium	500
Cadmium	500
Cobalt	500
Copper	500
Iron	500
Manganese	500
Nickel	500
Selenium	500
Strontium	500
Thallium	500
Zinc	500

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Rec'd 4-11-11 DE

SM-606-114 (TCLP-Spk)
Solution B

Lot # 1109508

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.98+%	HNO_3, 5%	10,000 µg/mL ± 0.5% Calcium Magnesium Potassium Sodium

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp Date: APR 07 2012
MSDS ATTACHED

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Certificate of Analysis

Rec'd 4-11-11 DE

SM-606-114 (TCLP-Spk) Solution C

Lot # 1109509

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts and Oxides	99.96+%	HNO_3, 2% HF, 0.5%	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Exp Date: APR 07 2012

Theodore C. Rains, Ph.D.
President

SM-606-114 (TCLP-Spk)

Solution C

Element List

(µg/mL)

Anitmony	500
Boron	500
Molybdenum	500
Silver	250
Tin	500
Titanium	500
Vanadium	250

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Certificate of Analysis

rec'd 4-11-12 OE

SM-606-114 (TCLP-Spk)
Solution D

Lot # 1109510

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.998+%	HNO_3, 5%	2500 µg/mL ± 0.5% Aluminum Arsenic Chromium Lead

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp Date: APR 07 2012
MSDS ATTACHED

Attachment 2

ICP: LCS & Matrix Spike Final concentration in solution (ug/L)		
ELEMENT	For Non-TCLP	For TCLP
Aluminum	2000	5000
Antimony	500	1000
Arsenic	2000	5000
Barium	2000	10000
Beryllium	50	1000
Cadmium	50	1000
Calcium	20000	20000
Chromium	200	5000
Cobalt	500	1000
Copper	250	1000
Iron	1000	1000
Lead	500	5000
Manganese	500	1000
Magnesium	20000	20000
Nickel	500	1000
Potassium	20000	20000
Selenium	2000	1000
Silver	50	500
Sodium	20000	20000
Thallium	2000	1000
Vanadium	500	500
Zinc	500	1000
Boron	500	1000
Molybdenum	500	1000
Tin	500	1000
Titanium	500	1000
Strontium	500	1000
Silicon	4000	4000
Sulfur	4000	4000

Attachment 3

ICPMS Matrix Spiking Solution, ICPMS LCS/SPK	
Element	**Matrix Spike Conc. (ug/L)**
Aluminum (Al)	2500
Antimony (Sb)	25
Arsenic (As)	50
Barium (Ba)	50
Beryllium (Be)	25
Boron (B)	500
Cadmium (Cd)	25
Calcium (Ca)	2500
Chromium (Cr)	50
Cobalt (Co)	25
Copper (Cu)	50
Iron (Fe)	2500
Lead (Pb)	25
Magnesium(Mg)	2500
Manganese (Mn)	250
Molybdenum (Mo)	50
Nickel (Ni)	50
Potassium (K)	2500
Selenium (Se)	50
Silver (Ag)	25
Sodium (Na)	2500
Strontium (Sr)	50
Thallium (Tl)	20
Tin (Sn)	50
Titanium (Ti)	50
Vanadium (V)	50
Zinc (Zn)	250

Title: Extraction of Pesticides and PCBs in Water by Separatory Funnel using SW846 Method 3510C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

06/27/19		06/27/19	
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06/27/19			06/27/19
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1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits:** SW846 Method 3510C describes a procedure for isolating organic compounds from aqueous samples, including concentration techniques suitable for preparing the extract for GC and/or GC/MS analysis. This SOP is specifically applicable to the isolation and concentration of water-soluble and slightly water-soluble Pesticides and PCBs using Method 3510C in preparation for analysis GC methods SW846 8081B, 8082A and GC/MS method EPA 680.

1.2. For a complete discussion of analytes and reporting limits (RLs) refer to the SOP for the applicable analytical method:.

- TestAmerica Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by SW846 Method 8081B,* current revision.
- TestAmerica Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by SW846 Method 8082A,* current revision.
- TestAmerica Edison SOP No. ED-MSS-010, *Determination of PCBs in Water and Soil/Sediment by GC/MS, EPA Method 680*, current revision.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 A measured volume of aqueous sample is spiked with surrogates and serially extracted with methylene chloride using a separatory funnel. The extract is dried by passing it through activated sodium sulfate, concentrated using a nitrogen blowdown technique and exchanged into hexane prior to analysis by Methods 8081B, 8082A or 680. Cleanup of extracts may be required prior to analysis.

2.2 The preferred option for preparing aqueous samples for analysis by SW8081B/8082A includes use of a reduced initial volume (~250ml) for analysis by a large volume injection (LVI). A technique for extraction of a larger volume (1000 ml) is also described.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Phthalate esters contaminate many products commonly found in the laboratory. Plastics, in particular, must be avoided, because phthalates are often used as

plasticizers and are easily extracted from plastic material. Phthalate contamination may result at any time if consistent quality control is not practiced.

Method interference may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interference under the conditions of the analysis, by analyzing method and reagent blanks.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggle when it is performed.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions (if applicable).			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Equipment

- Steam Bath - Fisher Scientific 66738 or equivalent.
- Muffle Furnace - Thermolyne Type 6000 or equivalent.
- N-Evap - Meyer Analytical Evaporator Model No. 112 or equivalent
- Separatory Funnel Rotator, APR Machine or equivalent
- Centrifuge, Varifuge F; Hereaus Sepatech

6.2. Supplies

- 400 ml Clear Glass Jar
- 500 ml Separatory Funnel, AMK Glass SFC-0095 or equivalent
- 100 mm o. d. glass funnels, Fisher or equivalent
- 10 ml jacketed, graduated Concentrator Tubes, AMK Glass KD-0018 or equivalent
- 19/22 Ground Glass Stoppers

- 50 ul Gastight Syringe, Hamilton 80900 or equivalent
- 100 ul Gastight Syringe, Hamilton 81000 or equivalent
- 150 ml Centrifuge Tube
- 100 ml Graduated Cylinder
- Pasteur 5¾" Disposable Pipettes, Fisher 13-678-20B or equivalent
- Glass Wool
- Desiccators
- Standard Taper Clamps (Size 19, blue)
- Wax Pencil
- 1 Liter Graduated Cylinder
- Marking Tags
- pH paper

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride and Acetone, is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

- Methylene Chloride - JT Baker Ultra-Resi 9254-03 or equivalent
- Sodium Sulfate Crystals – Mallinckrodt MA8024-06 or equivalent
- Acetone, J. T. Baker Ultra-Resi 9264-03 or equivalent
- Hexane, Pesticide Grade, Baxter 217-4or equivalent
- Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At TestAmerica Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).

7.1.1 Reagent preparation

7.1.1.1 Anhydrous sodium sulfate crystals (Mallinckrodt MA8024-06 or equivalent) must be baked in the muffle furnace for four hours at 400°C and serially rinsed with methylene chloride prior to use.

7.2 Standards

7.2.1 Standards are purchased as concentrated solutions (see Section 7.2.2). Most stock solutions are diluted (in volumetric glassware) to working concentration using acetone as the diluent as described in Section 7.3.

7.2.2 **Standard mixes and sources:** Table 1 lists the standard concentrate mix sources. Table 2 details the individual components of these mixes.

Table 1a:
Pesticide/PCB Standard Mixes and Sources (8081/8082)*

Standard Name ("Lab Name")	Concentration in ug/ml (each component)	Source - Catalog #
Pesticide Surrogate Spike Mix	200	RESTEK-32000
Aroclor Spike Mix (1660 Aroclor)	1000	RESTEK-32039
Organochlorine Pesticides Mix ("Pest Spike")	2000	RESTEK-32415
Chlordane (technical) ("Technical Chlordane Spike")	5000	RESTEK-32072
Toxaphene	5000	RESTEK-32071

*May be substituted with equivalent standards from alternate sources.

Table 1b:
PCB Homologue Standard Mixes and Sources (EPA 680)*

Standard Name ("Lab Name")	Concentration in ug/ml (each component)	Source - Catalog #
Retention Time Calibration Standard Mixture	Varies	Ultra-CB682-MN
Concentration Calibration Standard Mix	Varies	Ultra-CB681-MN
$^{13}C_{12}$-Decachlorobiphenyl Surrogate	40	Cambridge-EC1410-3
Lindane13C6	100	Cambridge-CL1282-S

Table 2a:
Components of Pesticide/PCB Standard Mixes (8081/8082)

Parameter	Catalog Nos.	Lab Standard Name	Conc (ug/ml)
Decachlorobiphenyl (DCB)	Pesticide Surrogate Spike Mix	RESTEK-32000	200
2,4,5,6-Tetrachloro-m-xylene (TCMX)	Pesticide Surrogate Spike Mix	RESTEK-32000	200
Aroclor 1016	Aroclor Spike Mix	RESTEK-32039	1000
Aroclor 1260	Aroclor Spike Mix	RESTEK-32039	1000
Aldrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
alpha-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
beta-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
Lindane	Organochlorine Pesticides Mix	RESTEK-32415	2000
g-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
DDD	Organochlorine Pesticides Mix	RESTEK-32415	2000
4,4'-DDE	Organochlorine Pesticides Mix	RESTEK-32415	2000

Table 2a: Components of Pesticide/PCB Standard Mixes (8081/8082)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Conc (ug/ml)**
4,4'-DDT	Organochlorine Pesticides Mix	RESTEK-32415	2000
Dieldrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
alpha-Endosulfan	Organochlorine Pesticides Mix	RESTEK-32415	2000
beta-Endosulfan	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endosulfan Sulfate	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin aldehylde	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin ketone	Organochlorine Pesticides Mix	RESTEK-32415	2000
Heptachlor	Organochlorine Pesticides Mix	RESTEK-32415	2000
Hetptachlor epoxide	Organochlorine Pesticides Mix	RESTEK-32415	2000
Methoxychlor	Organochlorine Pesticides Mix	RESTEK-32415	2000
Chlordane (technical)	Technical Chlordane Spike	RESTEK-32072	5000
Toxaphene	Toxaphene Spike	RESTEK-32071	5000

Table 2b: Components of PCB Homologue Standard Mixes (680)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Conc (ug/ml)**
PCB-104	Ultra-CB682-MN	Retention Time Calibration Standard Mix	100
PCB-208	Ultra-CB682-MN	Retention Time Calibration Standard Mix	200
PCB-77	Ultra-CB682-MN	Retention Time Calibration Standard Mix	100
Total Nonachlorobiphenyls	Ultra-CB682-MN	Retention Time Calibration Standard Mix	200
DCB Decachlorobiphenyl	Ultra-CB681-MN	Concentration Calibration Standard Mix	250
Total Dichlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Total Heptachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	150
Total Hexachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Monochlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Total Octachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	150
Total Pentachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Tetrachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Trichlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Decachlorobiphenyl-13C12	Cambridge-EC1410-3	$^{13}C_{12}$-Decachlorobiphenyl Surrogate	40
Lindane13C6	Cambridge-CL1282-S	Lindane13C6	100

7. 3. Standards Preparation

7.3.1. All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware. **Note:** septa on all surrogate and spike vials are to be replaced immediately after use. Additionally, all surrogate and spike vials are to be returned to the standards refrigerator immediately after use.

7.3.2. **Pesticide Spiking Standard (LCS/MS/MSD): For the reduced volume extraction option:** prepare a 4 ug/ml spiking solution by diluting 10 ml of the 20ug/ml standard prepared above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.3. **8082 PCB Spiking Standard (LCS/MS/MSD):**. **For the reduced volume extraction option:** prepare a 20 ug/ml spiking solution by diluting 10 ml of the 100 ug/ml stock standard prepared above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.4. **680 PCB Homologue Spiking Standard (LCS/MS/MSD):** The EPA Method 680 PCB homologue spiking standard is prepared by diluting 1.0 ml of the Retention Time Calibration Standard Mix (Ultra-CB682-MN) and 1.0 ml of the Concentration Calibration Standard Mix (Ultra-CB681-MN) to 100mL of hexane using volumetric glassware. For spiking instructions refer to Section 10.

7.3.5. **Technical Chlordane Spiking Standard (LCS/MS/MSD):** The technical Chlordane spiking solution is prepared at a final concentration of 100 ug/ml by diluting 0.2 ml of the 5000 ug/ml Technical Chlordane Spike solution (RESTEK-32072, see Tables 1 and 2) to a 10 ml final volume using acetone. For spiking instructions refer to Section 10.

7.3.6. **Toxaphene Spiking Standard (LCS/MS/MSD):** A Toxaphene spiking solution is prepared at a final concentration of 100 ug/ml by diluting 0.2 ml of the 5000 ug/ml Toxaphene Spiking Standard (RESTEK-32071, see Tables 1 and 2) to a 10 ml final volume with acetone. For spiking instructions refer to Section 10.

7.3.7. **Pesticide Surrogate Spiking Standard: For the reduced volume extraction option:** prepare a 2 ug/ml spiking solution by diluting 10 ml of the 10 ug/ml stock standard described above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.8. **680 PCB Homologue Surrogate Spiking Standard:** The EPA Method 680 surrogate spiking standard is prepared by diluting 3.125 ml of Decachlorobiphenyl-13C12 Surrogate (Cambridge-EC1410-3) and 1.0 ml of Lindane13C6 (Cambridge-CL1282-S) to 50.0 ml hexane using volumetric glassware. For spiking instructions refer to Section 10.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	Amber Glass	250 ml (8 oz)	Cool 4 ± 2°C	7 days to extraction; 40 days to analysis	USEPA SW846
Water	Amber glass	1000 ml	Cool 4 ± 2°C	7 days to extraction; 40 days to analysis	USEPA Method 680

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2°C) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample used for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria. If the spiked sample recovery results fall outside the laboratory generated limits (refer to the current active TALS

method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in the applicable analytical SOP (see Section 15.0, References).

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing DCB and TCMS (see Section 7.3.6). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

10.0. Procedure

10.1. Rinse a 500 ml separatory funnels twice each with a 1:1 methylene chloride/acetone mix and once with methylene chloride. Drain the solvents before adding sample to the funnels.

10.2. Place a small amount of glass wool into a 100-mm funnel and fill with pre-baked sodium sulfate crystals. Rinse three times with methylene chloride. Also rinse the outside of the funnel stem three times with methylene chloride, as it is likely to come into contact with the extract. Allow time for all of the rinsate to drain out of the funnel into a waste container.

10.3. Write the lab sample id number for each sample on the separatory funnels using a red wax pencil.

10.4. Make up hangtags for each sample as follows and hang on the corresponding

PCBs	PST
Sample Number: xxx	Sample Number: xxx
Fraction-Matrix:xxx	Fraction-Matrix:xxx
Date of Extraction:xx/xx/xx	Date of Extraction:xx/xx/xx

10.5. Place a 100 ml funnel containing rinsed sodium sulfate crystals onto each flasks

10.6. Mark the fluid level on each sample bottle with a black magic marker.

10.7. Rinse out a 1000ml graduated cylinder two to three times with organic free reagent water (for TCLP/SPLP leachates or reduced volume extraction use a 250 ml graduated cylinder). Obtain 1000ml of organic free reagent water (100 ml for TCLP/SPLP leachates or 250ml for reduced volume extraction) from the Millipore filtering apparatus for each of the method blank and the LCS.

10.8. Measure the initial pH of each sample with wide range pH paper and record the observed value in TALS prep batch as 'Received/initial pH'. If the initial pH of the sample is not between 5-9 adjust the pH to within 5-9 using 1:1(v/v) sulfuric acid or 10N Sodium hydroxide, as appropriate. Record the final adjusted pH value in the TALS batch comment section. Lesser strengths of acid or base solution may be employed, provided that they do not results in a significant change (<1%) in the volume of sample.

10.9. Pour each sample (including the method blank and LCS water) into its corresponding separatory funnel.

10.10. Rinse the spiking syringes 8 to 10 times with Acetone.

10.11. **For methods 8081/8081**: Add 50 ul of Pesticide Surrogate Spike Mix (see Section 7.2) to each sample and QC sample. When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel

10.12. **For method 680**: add 1.0 ml of the PCB Homologue Surrogate Spiking Standard (see Section 7.3.8) to each sample and QC sample. When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel

10.13. Depending upon the fraction and requested target analytes (Refer to Section 7.3 for information of spiking solutions).

- **PCBs by 8082**: add 50 ul of the 20ug/ml PCB Spike solution (see Section 7.3.3) to each LCS and designated MS/MSD
- **Pesticides (total) and TCLP/SPLP Pesticides by 8081**: 50 ul of the 4 ug/ml Pest Spike solution (see Section 7.3.2) to each LCS and designated MS/MSD
- **Technical Chlordane by 8081:** 20 ul of the 100 ug/ml Technical Chlordane Spike mix (see Section 7.3.5) to each LCS and designated MS/MSD.
- **Toxaphene by 8081:** 20 ul of the 100 ug/ml Toxaphene Spike mix (see Section 7.3.6) to each LCS and designated MS/MSD.

- **PCB Homologues by 680:** 1ml of the 680 PCB Homologue Spiking Standard (see Section 7.3.4) to each LSC and designated MS/MSD).

When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel. Add 15 ml of methylene chloride for the reduced volume extraction option.

10.14. Add 15 ml of methylene chloride to each sample bottle. Swirl the solvent in the bottle and add the 15 ml for of methylene chloride to its corresponding separatory funnel.

10.15. Secure the separatory funnels to the rotators. Start the rotators.

10.16. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.17. After the rotator stops let the sample settle.

10.18. Drain bottom layer (organic layer) of the sample into the funnel/Concentrator apparatus.

10.19. Repeat steps 10.12 through 10.16 twice, now adding the methylene chloride directly to separatory funnel, rather than rinsing the sample container as in 10.12.

10.20. If an emulsion forms during the extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the large separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride with the desired sample extract on the bottom. A 1-ml disposable pipette should be used to transfer the bottom layer from the centrifuge tube to the appropriate concentration tube. With this method, care must be taken not to transfer any of the top layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.21. Find the empty sample bottles previously marked with a black marker with tap water up to the black line. Pour this into the graduated cylinder used for measuring sample volumes. Record each sample volume into the TALs extraction batch database.

10.22. Rinse funnel/sodium sulfate attached to concentration tube, twice with about 20ml of methylene chloride.

10.23. Sample can be concentrated between 30°C-40°C in the N-Vap to 2 ml final volume in the methylene chloride

10.24. Add about 10ml of Hexane to extract to exchange

10.25. Concentrate the hexane extract to 1 ml on the steam bath (temperature set between 30°C-40°C) using the N-EVAP apparatus.

10.26. The extract is now ready for the appropriate clean up.

10.27. If an emulsion forms during extraction, rinse a centrifuge tube well with Methylene Chloride and then drain the lower layer from the large separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the Methylene Chloride with the desired sample extract on the bottom. A 1-ml disposable pipette should be used to transfer the bottom layer from the centrifuge tube to the appropriate concentration tube. With this method, care must be taken not to transfer any of the top layer. The top layer that remains is poured back into the 2000-ml separatory funnel with the rest of the original sample.

10.28. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.28.1. The TALS reagent database must be completed in full with all required information whenever standards are logged and/or prepared.

10.28.2. All required data types (sample volumes, reagent and standard volumes, reagent and standard lots, spike witness, etc…) must be entered into the TALS batch database each time an extraction is performed. The department manager is responsible for ensuring that the data is reviewed for completeness and accuracy.

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11.0. Calculations / Data Reduction

None

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a

calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to the current revision of TestAmerica Edison SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½

inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method 3510C: Separatory Funnel Liquid-Liquid Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. *EPA Method 680: Determination of Pesticides and PCBs in Water and Soils/Sediment by Gas Chromatography/Mass Spectrometry*. Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, USEPA, Cincinnati, OH, November 1985.

15.3. TestAmerica Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by SW846 Method 8081B,* current revision.

15.4. TestAmerica Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by SW846 Method 8082A,* current revision.

15.5. TestAmerica Edison SOP No. ED-MSS-010, *Determination of PCBs in Water and Soil/Sediment by GC/MS ,EPA Method 680*, current revision.

15.6. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.7. TestAmerica Corporate Document No. CW-E-M-001, *Environmental Health and Safety Manual,* current revision.

15.8. TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.9. TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.10. TestAmerica Edison SOP No. ED-GEN-013, *Glassware Cleaning*, most current revision

15.11. TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision.

16.0. Method Modifications:

NONE

17.0. Attachments

NONE

18.0. Revision History

- Revison 13, dated Jun 27, 2019:
    - Sections 1 and 15 (and throughout document as applicable): Deleted references to obsolete methods SW8082 and SW8081A as well as references to associated SOPs.
    - Section 2.2: revised wording to clarify that the RVE/LVI technique is the standard procedure.
    - Throughout document: removed references to equipment no longer used in the procedure: Synder columns, Kuderna-Danish concentrators, Erlenmyer flasks.

- Revision 12, dated May 16, 2017:
    - Updated throughout to include aqueous preps for EPA method 680.
- Revision 11, dated Dec 13, 2016:
    - Section 6.2: added pH paper to list of supplies.
    - Section 10.8: added instruction for measuring, adjusting and documenting sample pH values.
- Revison 10, dated Jun 15, 2015:
    - Section 2.1 and throughout document: Revised standard initial volume for Reduced Volume Extraction (RVE) to 250 ml.
    - Section 7.2: Tables 1 and 2 updated with new Restek standards.
    - Sections 7.3: updated spiking standards preparation instructions as necessitated by switch to Restek standards.
    - Section 8.0: updated minimum volume to 250ml.
- Revison 9, dated May 3, 2013:
    - Section 7.2.2, Table 1: corrected concentration of the Chlordane (technical) standard solution to 1000 ug/ml. Deleted 'TCLP Pest Mix 1' and 'TCLP Pest Mix 2'. Added Toxaphene (Supleco 48103-U).
    - Section 7.2.2, Table 2: corrected concentration of the Chlordane (technical) standard solution to 1000 ug/ml. Deleted all TCLP Pest Spike standards. Added Toxaphene (Supleco 48103-U).
    - Deleted Section 7.3.4, TCLP Pesticide Spiking Standard (LSC/MS/MSD). Remaining sections renumbered accordingly.
    - Section 10.11. Deleted last bullet (TCLP Pesticides). Added '…and TCLP/SPLP Pesticides' to the second bullet (Pesticides). Added '(or 20 ul of the 100ug/ml mix for reduced volume extraction)' to the Toxaphene bullet.
- Revision 8, dated February 26, 2013:
    - Throughout document: updated Lab Quality Manual section number references as required.
    - Section 1.2: added references to all applicable analytical SOPs.
    - Section 2.0: added Section 2.1 which describes option for Reduced Volume Extraction.
    - Section 6.2: added a 500 ml separatory funnel.
    - Section 7.0: updated throughout to include additional standards preps for reduced volume extraction.
    - Section 8.0: updated to include smaller sample container for the reduced volume extraction option.
    - Section 10.0: updated to include spiking and concentration instructions for the reduced volume extraction option.

- Revision 7, dated October 31, 2011

    - Section 7.0: added 'Reagent Water' and details concerning it's preparation to the list of reagents.

- Revision 6, dated October 30, 2009

    - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
    - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
    - Section 1.1: Expanded to include references to applicable analytical SOPs.
    - Section 3: revised to reference new location for definitions.
    - Section 5: Revised to include most up to date corporate health and safety references and information.
    - Section 7.2.2: Added tables detailing components found in the various spiking standards mixes.
    - Section 7.3: Updated the instructions for preparation of spiking standards.
    - Section 7.3: Added tables with spiking standards prep details.
    - Section 8: Updated with additional details including a table outlining containers, preservation and holding times.
    - Section 9.1: Expanded QC sample preparation details.
    - References: Expanded to include more specific SOP references
    - Section 18: Added this Revision History section
    - Throughout document: added references to TestAmerica LIMS (TALS).
    - Throughout document: deleted references to organophosphorus pesticides as the Edison lab no longer analyzes for these.

Title: Extraction of Semi-Volatile Organic Compounds in Aqueous Samples and Leachates - Separatory Funnel, SW846 Method 3510C

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits** SW846 Method 3510C describes a procedure for isolating semivolatile organic compounds from aqueous samples and leachates, including concentration techniques suitable for preparing the extract for GC/MS analysis. This SOP is applicable to the isolation and concentration of water-soluble and slightly water-soluble semivolatile organics in preparation for analysis by SW846 Methods 8270C or 8270D.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to TestAmerica Edison SOP Nos. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270C*, current revision and ED-MSS-009, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270D*, current revision

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. A measured volume of sample (~250 mL) is serially extracted with methylene chloride at a pH less than 2 and again at a pH greater than 11 using separatory funnel extraction. The methylene chloride extract is dried and concentrated to a volume of approximately 2 mL. Nitrogen blowdown is employed as the final concentration step. The extract is subsequently analyzed by SW846 Method 8270C or 8270D (GC/MS) by a large volume injection (LVI) technique. This procedure is referred to throughout as Reduced Volume Extraction (RVE) and Large Volume Injection (LVI).

2.2. An option for preparing aqueous samples using a larger initial volume (~1000 ml) is also described. This procedure is referred to as 'large volume' throughout the document.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Solvents, reagents, glassware, and other sample hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.2. Phthalate esters contaminate many products commonly found in the laboratory. Plastics, in particular, must be avoided, because phthalates are often used as plasticizers and are easily extracted from plastic material.

Phthalate contamination may result at any time if consistent quality control is not practiced.

4.3. The decomposition of some analytes has been demonstrated under basic extraction conditions. Phthalate esters may exchange and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by shorter reaction times. Performing the initial extraction at an acid pH will optimize the recovery of phenols

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted, periodic venting may be necessary during the extraction. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, the use of a face shield over safety glasses or goggles is recommended. Keep the sash on the fume hood as low as reasonably possible.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in

the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Separatory funnel rotator, APR Machine or equivalent
- Analytical Evaporator (N-Evap) Organomation
- Centrifuge, Varifuge F; Hereaus Sepatech
- Six Position Steam Bath, Fisher 15-496 or equivalent

6.2. Supplies

- 250 ml Erlenmeyer Flask, AMK Glass ERL-0252 or equivalent
- 2000 ml or 500 ml Separatory Funnel, AMK Glass SFC or equivalent
- 100 mm o. d. glass funnels, Fisher or equivalent

- 10 ml jacketed, graduated Concentrator Tubes, AMK Glass KD-0018 or equivalent
- 19/22 Ground Glass Stoppers
- 3 Ball Snyder Columns, TEC Glass TG6-03 or equivalent
- 1 ml Gastight Syringe, Hamilton 81317 or equivalent
- 150 ml Centrifuge Tube
- 100 ml Graduated Cylinder
- Pasteur 5 ¾" Disposable Pipets, Fisher 13-678-20B or equivalent
- Kuderna Danish Flask (500 ml), TEC Glass TG7-01 or equivalent
- Vials, 2ml amber screw cap with Teflon liner
- Glass Wool
- Dessicator
- Standard Taper Clamps (Size 19, blue)
- Boiling Stones, Troemner P/N 133-B or equivalent, rinsed with Methylene Chloride
- pH Paper
- Watch Glass
- Wax Pencil
- 1 Liter Graduated Cylinder
- Marking Tags

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.1.1 Methylene Chloride – JT Baker Ultra-Resi 9254-03 or equivalent

7.1.2 Acetone, J.T. Baker Ultra-Resi 9264-03 or equivalent

7.1.3 Methanol, J.T. Baker, Pesticide Grade, 9077-02 or equivalent

7.1.4 Concentrated Sulfuric Acid - Baxter 2876-9 or equivalent

7.1.5 Sodium Hydroxide Pellets – Baxter 7708-500NY or equivalent

7.1.6 Sodium Sulfate Crystals – Mallinckrodt MA8024-06 or equivalent (Must be baked in the muffle furnace for four hours at 400°C and serially rinsed with Methylene Chloride prior to use.)

7.1.7 Sodium Hydroxide (10 N) - Fill a precleaned 1000 ml volumetric flask with 500 mls deionized water. Weigh out 400 g NaOH pellets and dispense slowly into the flask. Stir slowly until the pellets dissolve, then add more deionized water until the 1000 ml level is

reached. Be careful as this procedure generates heat. Never add water to the reagent that is to be dissolved.

7.2 Standards

7.2.1 Most stock target analyte standard solutions are purchased as prepared solutions; other standards are prepared in the laboratory using neat compounds (see table below). Most stock solutions are diluted (in volumetric glassware) to working concentration using methylene chloride as the diluent as described below. Stock standards of similar quality from other suppliers may be substituted as required.

NOTE: The standards listed here are used as calibration standards and spiking standards. Separate source calibration verification standards are addressed in the analytical SOPs.

Standard Name	Concentration	Vendor	Catalog #
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	RESTEK	570666
8270 List 1/ Std#10Benzoic Acid	2000ppm	RESTEK	569731
8270 List 1/ Std#9	2000ppm	RESTEK	569730
Custom SVO Mix	2000ppm	SPEX	SVO-TANJ-16-5
Bisphenol A	1000ppm	SPEX	S-509-MC
8270 List 1/ Std#11	2000ppm	RESTEK	569732
8270 Surrogate Standard	5000 ppm	RESTEK	567685
Aromatic Amines Custom Mix	2000 ppm	Supelco	21467482

7.2.1.1. **Spiking Standard:** For use in spiking aqueous samples including TCLP/SPLP leach being prepared for BNA analysis by SW846 Method 8270. Prepare the second source spiking solution for MS/MSD/Blank Spike (LCS) as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	20 ml (methanol)	50/100/200 ppm
8270 List 1/ Std#10	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/	2000ppm	10 ml (methanol)	100 ppm

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
Std#9			
Custom SVO Mix-SPEX	2000ppm	10 ml (methanol)	100 ppm
Bisphenol A-SPEX	1000ppm	10ml (methanol)	50ppm
8270 List 1/ Std#11	2000ppm	20ul (methanol) 100ul(methanol)	Used neat for LVI Used neat for non-LVI
Aromatic Amines Custom Mix**	2000ppm	10 ml (methylene chloride)	100 ppm

*** As needed based on clients requirement.*

Note: Neat spiking/surrogate standards are stored per vendor requirements (either room temp or 4 deg C, as appropriate). All prepared standard solutions are refrigerated at 4 deg C.

7.2.2 **8270 Surrogate Standard Spiking Solution**: For use in spiking all blanks, samples and associated QC prior to extraction. Prepare as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 1000 ml methanol	Final Concentration
8270 Surrogate Standard	5000 ppm	20 ml	100 ppm

7.2.3 Internal standard is prepared added by the analytical department. For details see analytical SOPs No. ED-MSS-002 (SW846 8270C) or ED-MSS-009 (SW846 8270D).

7.2.4 The preparation of all standards must be documented in TestAmerica LIMS (TALS) or a standard preparation logbook. Information such as standard supplier, lot number, original concentration, and a description of how standard was prepared are required along with a laboratory lot number, analyst's initials, date prepared and verification signature. Standards must be made every 6 months or sooner if signs of degradation appear.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

8.1 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.2 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.3 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water or Leachate	250ml Amber (RVE)/ Amber glass, 1L	250ml-RVE/ 1000 ml	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270C

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples. Refer to TestAmerica Edison analytical SOPs No. ED-MSS-002 (SW846 8270C*)* or ED-MSS-009 (SW846 8270D) for details on analysis and evaluation of these QC elements:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are updated annually and are updated into lab reporting software.

9.1.1. **Method blanks** are extracted with every sample batch on each day that samples are extracted.

9.1.2. **Matrix Spike (MS)/Matrix Spike Duplicate (MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. (Note: an

LCS/LCSD may be substituted for the MS/MSD if insufficient client environmental sample volume is available).

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.2).

10.0 Procedure

NOTE: The sample preparation procedure for aqueous samples (Section 10.1) contains two options: reduced volume extraction (RVE) (250ml) for large volume injection (LVI) analysis and large volume extraction (1000ml),

10.1. Sample Preparation for Aqueous/ Leachates Samples

10.1.1. Rinse the required number of 500-ml separatory funnels (when RVE is required) or 2000-ml separatory funnels (when large volume extraction is required) and 250-ml Erlenmeyer flasks twice with a 1:1 mixture of Methylene Chloride:Acetone and once with Methylene Chloride.

10.1.2. Place a small amount of glass wool into a 100-mm funnel and fill with pre-baked sodium sulfate crystals. Rinse three (3) times with Methylene Chloride. Also rinse the outside of the funnel stem three (3) times with Methylene Chloride (since the stem is likely to come into contact with the extract). Allow time for all of the rinsate to drain out of the funnel into a waste container.

10.1.3. Record the lab sample numbers on the separatory funnels with red wax pencil.

10.1.4. Make up tags with the following information and place on a 250ml Erlenmeyer flask:

BNAs		**BNs**	**AEs**
Acid Fraction	BN Fraction		
Sample Number	Sample Number	Sample Number	Sample Number
Fraction-Matrix	Fraction-Matrix	Fraction-Matrix	Fraction-Matrix
Date of Extraction	Date of Extraction	Date of Extraction	Date of Extraction

10.1.5. Place the 100ml funnel containing rinsed sodium sulfate crystals onto the flask.

10.1.6. Mark the fluid level on the sample bottles with a black Sharpie. Pour each sample into its corresponding separatory funnel. Fill each sample bottle to the black line with tap water. Pour this into the graduated cylinder used for measuring sample volumes. Note the volume for each sample on the Organic Extraction Data Sheet.

10.1.7. Rinse out a graduated cylinder with lab reagent water two to three times. Using the graduated cylinder obtain 250 ml when RVE is used (or 1000-ml when large volume is required) of lab reagent water from the Millipore filtering apparatus located in the Wet Chemistry laboratory for each of the method blank and the laboratory control sample (LCS) (aka blank spike).

10.1.8. Pour each the reagent water for the method blank and LCS into the corresponding separatory funnels.

10.1.9. Rinse syringes eight (8) to ten (10) times with Methylene Chloride.

10.1.10. If you are performing BNAs add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11. If you are performing *BN only* extraction, add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.20. If you are performing *AE only* extraction add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11.

10.1.10.1. If extracting QC samples (MS, MSD, LCS or LCSD), add 0.2 ml when RVE is used (or 1.0 ml when large volume is required of the Spiking (see Section 7.2.1.1) to the appropriate separatory funnel. Note: When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Do not touch the tip of the syringe to the liquid or the side of the separatory funnel.

10.1.11. Add concentrated sulfuric acid to each sample to adjust the pH to <2. (Usually you only need to add 1ml using small disposable Pasteur pipette). Note: pH adjustments must be documented in the extraction log.

10.1.12. Shake each sample for a short time and check pH using pH paper. The pH must be 2 or less. If the pH has not been lowered sufficiently, add more acid.

10.1.13. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to each sample bottle.

10.1.14. Swirl the bottle and add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to its corresponding separatory funnel.

10.1.15. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.16. Stop the rotation and allow the sample to settle.

10.1.17. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.18. Repeat steps 10.1.13 through 10.1.17 twice, adding the Methylene Chloride directly to separatory, rather than rinsing the sample container as in 10.1.15.

10.1.18.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.19. If you are preparing the sample for acid extractables analysis only, you are finished and you can now discard the remaining liquid in each separatory funnel and proceed to Section 10.1.27. If you are required to extract base/neutrals, proceed with Section 10.1.20.

10.1.20. Adjust the pH of the sample to >11 by adding 10N sodium hydroxide (NaOH) to the sample in each separatory funnel. NOTE: pH adjustment must be documented in the extraction logbook.

10.1.21. Shake each separatory funnel for a short time and check pH. It should be basic, >pH 11. If the pH is not as high as it should be, add more 10N sodium hydroxide (NaOH).

10.1.22. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required)

10.1.23. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.24. Stop the rotation and allow the sample to settle.

10.1.25. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.26. Repeat steps 10.1.22 through 10.1.25 twice.

10.1.26.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.27. Pour the entire methylene chloride extract from the Erlenmeyer flask into a KD concentration tube apparatus (pre-rinsed three times with acetone). Rinse the Erlenmeyer flask from which the sample came twice with methylene chloride and pour both rinsates into the KD apparatus.

10.1.28. Attach Snyder column (pre-rinsed three times with acetone) to the top of the KD apparatus.

10.1.29. For RVE extractions blow the extract directly down to a final volume of 2-ml on the N-Evap. Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 2.0 ml.

10.1.30. For large volume (1000 ml) extractions concentrate the extract to approximately 5-ml in Steam Bath Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 1.0 ml. Bring the volume of each extract up to 2ml with methylene chloride

10.1.31. Split each extract into two (2) -1ml aliquots and transfer each aliquot to a separate 2ml amber screw cap vial with Teflon liner. Label one vial as 'SIM" and the second vial as 'Total'. Transfer custody of the vials to the Semivolatile GC/MS laboratory for analysis (see TestAmerica Edison analytical SOPs No. ED-MSS-002 (SW846 8270C*)* or ED-MSS-009 (SW846 8270D).

10.2. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.2.1. The Standards Prep Logbook or TALS Reagent Database must be completed in full with the required information whenever standards are logged and/or prepared.

10.2.2. Each time an extraction is performed, the applicable TALS data record must be completed and reviewed the Organic Prep Supervisor or designee. Lot numbers of all reagents and solvents used or added to samples during preparation must be documented in the database.

10.2.3. Each sample extracted must be included in a batch and be recorded in the TALS database.

10.2.4. Following the extraction procedure, the technician must complete all TALS data fields pertaining to the samples extracted.

11.0. Calculations/Data Reduction

n/a

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1 United States Environmental Protection Agency, “Method SW3510C, Separatory Funnel Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2 TestAmerica Edison SOP No. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS, SW846 Method 8270C*, current revision.

15.3 TestAmerica Edison SOP No. ED-MSS-009, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270D*, current revision

15.4 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.5 TestAmerica Environmental Health and Safety Manual, CW-E-M-001.

15.6 TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*), current revision.

15.7 TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.8 TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision

15.9 TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)*

15.10 TestAmerica Edison SOP No. ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).*

16.0. Method Modifications:

N/A

17.0. Attachments

N/A

18.0. Revision History

Revision 12, effective 03/26/2018

- Updated throughout to clarify that the lab's standard procdure is to use Reduced Volume Extraction (~250 ml initial volume) with an option to use large volume extraction (~1000 ml initial volume).

Revision 10, effective 11/29/2016:

- Sections 7.2.1 and 7.2.1.1 : updated current sources of standards.
- Section 7.2.1.1: added standards storage information as note at end of section.
- Section 8.0: added option for 250 ml amber sample containers (LVI option)

Revision 9, effective 11/21/2014:

- Section 7.2: updated current sources of all standards.
- Throughout document as required: added option for preparation of leachates by LVI.
- Section 10.1.29 through 10.1.31: clarified the concentration techniques for both full volume and LVI extracts.

Revision 8, effective 11/28/2012

- Throughout document: updated references to Lab Quality Manual section numbers.
- Added references as necessary throughout to TestAmerica Edison SOP No. ED-MSS-009 (*Semivolatile Organic Compounds by GC/MS, SW846 Method 8270C*D, current revision.
- Section 2.2 added describing option for analysis of lower initial volume for subsequent analysis using large volume injection (LVI) technique.
- Section 6.2: added 500 ml separatory funnel.
- Sections 7.2.1 and 7.2.1.1: added '5 Compound BNA Custom Mix' and 'Aromatic Amine Custom Mix' to list of standards and prep instructions table.
- Section 10.1 (Sample Prep for Aqueous Samples): revised throughout to include option for extraction of reduced aqueous volume (250ml) for subsequent analysis by LVI technique. Added note as preface to Section 10.1 alerting analyst to the two available extraction volume options (1000ml and 250ml).

Revision 7, effective 12/6/10

- Section 3: revised to reference new location for definitions

- Sections 7.2.1 and 7.2.1.1: Added 4 compounds and additional details to the description of standard preparation of the Spiking Standard.
- Section 7.2.4: added option to document standards preparation within TALS rather than a laboratory notebook.
- Section 10.3: revised to include TALS as the main repository for raw data associated with sample prep.

Revision 6, effective November, 2008

- Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
- Revised title to include 'Leachates'.
- Section 1.3: Added reference to Quality Assurance Manual for method modifications.
- Section 3: revised to reference new location for definitions.
- Section 5: Revised to include most up to date corporate health and safety references and information.
- Section 7: added details of the solvent testing and approval program.
- Section 7.2.1: Added additional details to the description of standards and the preparation of the Spiking Standards and Surrogate Standards. Removed references to the Internal Standard which is now added by the analytical group and is discussed in TestAmerica Edison SOP No. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW846 Method 8270C*, current revision.
- Section 9: Quality Control: added additional details to the discussion of the various QC sample types
- Section 10: Revised and clarified to reflect current procedures. Removed reference to internal standard addition (now completed by analytical group).
- Section 11: Removed reference to Organic Calculation SOP.
- Section 12: updated and revised the MDL requirements to reflect text in the current revision of the TestAmerica Edison Laboratory Quality Manual (LQM).
- Section 15: References: Expanded to include more specific SOP references
- Section 16: Added Section 16 (Method Modifications).
- Section 18: Added this Revision History section

Title: Purge and Trap for Aqueous Samples, SW846 Method 5030B and 5030C

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method describes purge and trap techniques for the analysis of volatile organic compounds in aqueous samples by SW846 Method 5030B and 5030C and high concentration soil sample extracts prepared using SW846 Methods 5035 or 5035A.

1.1.2 SW846 Methods 5030B and 5030C can be used for most volatile compounds that have boiling points below 200° C and are insoluble or slightly soluble in water. Water-soluble compounds can be analyzed for by this method; however, quantitation limits will be higher due to poor purging efficiency.

1.1.3 This sample introduction step is to be followed by determinative methods such as SW-846 Method 8015 for GC analysis or SW-846 8260 for GC/MS analysis.

1.1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 9 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 An aliquot of the aqueous sample is purged with helium (or nitrogen, where applicable) as in a closed sparging vessel. For medium or high level samples, an aliquot of the extract prepared by SW846 Methods 5035 or 5035A is combined with organic free reagent water and then purged. The volatile compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is complete, the sorbent trap is heated and backflushed with helium (or nitrogen, where applicable) to desorb the volatiles onto a gas chromatographic column.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method and the determinative methods that follow are susceptible to contamination from a number of sources. Potential sources of contamination include organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum

during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatile laboratory is housed in a separate building, away from the organic extraction area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatile laboratory.

4.3 The helium (or nitrogen, where applicable) used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 A trip blank prepared from organic-free reagent water is carried through the sampling, storage, and analysis of each group of samples to check for such contamination.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Purge vessels are removed from the autosampler units after each use, rinsed, baked, returned to the units and pre-purged before the next use.

4.7 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.8 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

- Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

- Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

Purge and trap unit that consists of three parts: the sample purger, the trap, and the concentrator.

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required.

6.1.2 Purge and trap units used include the the OI 4451 autosampler/4560 concentrator, the Archon 5100A automatic sampler/OI 4560 concentrator, OI 4551A autosampler/4660 Concentrator, Centurion

Autosampler/Encon, Archon/Encon, Solatek autosampler/Stratum, Aquatek 70/Tekmar 3000 concentrator and Archon/Evolution.

6.1.3 The purge chambers of each unit are designed to accept a 5mL sample with a water column at least 3cm deep. The headspace above the water has a volume less than 15 mL. The purge gas is introduced no more than 5 mm from the base of the water column. The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3mm at the origin.

6.1.4 Different sorbent traps are used, depending on the determinative method that follows the purge and trap procedure. Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 The VOCARB 3000 (Supelco) is used in the Encon, Tekmar 3000 and Evolution. This trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001.

6.1.6 The OI Analytical purge trap #10 is used in the OI 4560 and 4660. This trap is 25cm long and has an inside diameter of 0.105 inches. The trap is packed to contain the following adsorbents: Tenax/silica gel/ carbon molecular sieve.

6.1.7 The concentrator of each unit is capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.8 Gas chromatograph. HP 5890/Agilent 6890N/7890 equipped with temperature programming capability.

6.1.9 GC column:

- 75M long x 0.53mm ID, J&W DB-624 capillary column with 3um film thickness or similar phase.
- 20M long x 0.18 ID, DB-624 or Rtx-VMS with 1 um film thickness.
- 30M long x 0.25 mm ID x 1.4 um film thickness Rtx-624.

6.1.10 Injection port liners. HP 18740-80200 or equivalent.

6.2 Supplies

- Microsyringes. 10 uL to 1000 uL.
- Syringes. 5mL and 30mL gas-tight.
- Volumetric flasks. Class “A” glassware, 10mL, 50mL, and 100 mL.

- Vials. 2mL amber glass with screw cap with Teflon-faced septa.
- VOA vials. 40ml glass with PTFE-faced septa

7.0 Reagents and Standards

7.1 Organic free reagent water. Distilled water purchased from Poland Spring.

7.2 Methanol: Purge and trap grade, purchased from JT Baker. (Cat # 9077-02)

7.3 Standard preparation and use is described in detail in each of the determinative methods.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Aqueous samples are collected in the appropriate containers and preserved according to the determinative method requirements.

8.2 All aqueous samples are protected from light and stored at 4°C in an area free of organic solvents. No standards or solvents are stored in the sample refrigerators or in the surrounding area.

8.3 All aqueous samples are analyzed within 14 days of collection.

8.4 The collection, preservation and hold times for soil samples prepared via SW846 Method 5035 or 5035A are addressed in the corresponding TestAmerica Edison SOPs (see references in Section 15).

9.0 Quality Control

9.1 Specific quality control procedures are outlined in each of the determinative methods that follow this purge and trap technique of sample introduction. Standard quality assurance practices are used with all methods.

9.2 An initial demonstration of accuracy and precision is performed for each determinative method. This demonstration is done for each sample introduction technique.

9.3 Reagent water blanks are analyzed to ensure that reagents and/or sample dilutions are free of interferences.

9.4 Method blanks are prepared and analyzed in the same manner as samples, and are carried through the entire analytical procedure to show that each system is in control.

9.5 Each analysis batch contains spike and duplicate data as outlined in the method, as well as a laboratory control sample (blank spike) to show that replicate data can be generated and appropriate concentrations of target analytes recovered.

10.0 Procedure

10.1 This section provides guidance for the analysis of aqueous samples and/or methanol extracts prepared by SW846 Method 5035 by purge and trap techniques. Determinative methods SW846 Methods 8015 or 8260 will follow this purge and trap procedure

10.2 Instrument operating parameters are set at the beginning of a method of analysis and remain constant throughout the calibration and sample analysis steps. See each determinative method to verify specific instrument operating parameters.

Table 1: Example Instrument Operating Parameters

Purge and trap unit	
Purge Time	11 minutes
Dry Purge	1 Minutes
Purge Gas	Helium (or nitrogen, where applicable)
Purge Flow	40-45 ml/min
Purge Temp	Water: Ambient, Solids: 40^{0}C
Trapping Temp	Ambient, <30^{0}C
Desorb Time	1 Minute
Desorb Temp	VOCARB: 260^{0}C
Gas chromatograph	
Injector	180^{0}C
Carrier Gas	Helium
Carrier Flow	6 ml/min
Oven Program	35 - 250^{0}C with 2 ramps
Run Time	26 Minutes
Mass Spectrometer	
Electron Energy	70 volts (nominal)
Mass range	35-260 AMU
Scan time	0.9 sec./scan
Source Temp	200^{0}C
Separator Temp	180^{0}C

10.3 Screen all samples according to TestAmerica Edison SOP No. ED-GCV-001, (*Screening for Volatile Organics, Static Headspace with GC FID,, SW846 Method 5021* **,** current revision). Analysts are furnished with approximate dilution factors prior to analysis.

10.4 Allow all samples and standards solutions to warm to ambient temperature before use.

10.5 Prior to performing any purge and trap analysis, prepare each system in the following manner:

10.5.1 Condition concentrators by baking the trap at their bake temperatures for 5-12 minutes. Also condition the GC column by baking at 200°C.

10.5.2 Access the FLUSH menu on the Archon Autosampler. Rinse the syringe barrel with two 6mL portions of rinse water. Turn off the helium switch, and fill the water reservoir with reagent water. Visually inspect the standard vial and fill with the appropriate internal standard/surrogate mix. Turn the helium switch back on. Prime the standard loop by accessing the SYSTEM/MAINTENANCE/STANDARD CONTROL menu. Empty the waste reservoir.

10.5.3 Prepare other referenced autosamplers by filling internal standard reservoirs with the appropriate IS/SURR, empty the waste reservoir and fill the water rinse reservoir.

10.6 Prior to using this sample introduction technique, the GC or GC/MS system must be tuned and calibrated as required by each of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15). All method requirements for tune, calibration, and blank analysis must be met prior to sample analysis.

10.7 Aqueous samples

10.7.1 Load the sample vials directly into the autosampler tray. The appropriate internal standard and/or surrogate solution will be added to the sample by the autosampler unit immediately before a sample is purged.

10.7.2 The process of using autosamplers also destroys the validity of the sample by puncturing the septum. Any re-analysis should be done using second vial of sample. If only one vial exists, do not use this autosampler.

10.8 Medium or high level sample

10.8.1 Prepare samples by adding the appropriate amount of sample to a 50mL volumetric flask containing reagent water.

10.8.2 Add the appropriate amount of sample to the flask. Dilute to volume with reagent water, cap, and invert three times. Pour the diluted sample into a 5mL syringe or a 40mL VOA vial.

10.8.3 If a dilution is required, reduce the volume of sample added to the

10.8.4 Load sample onto the autosampler.

10.8.5 Purge the sample for 11 minutes, with one minute dedicated to removing moisture from the purge and trap system (dry purge). Times may vary among different determinative methods.

10.8.6 After purging is complete, desorb the sample onto the GC column by rapidly heating the trap to 260°C and backflushing it with helium (or nitrogen, where applicable). (Trap temperatures will vary based on type used)

10.8.7 Begin the GC temperature program and data acquisition.

10.8.8 Re-condition the trap by baking at 260°C for 8-12 minutes.

10.8.9 Cool the trap to (<31°C). The trap is now ready for the next sample.

10.8.10 Process data using Chrom software, and evaluate as per each determinative method.

10.9 Technical acceptance criteria for sample analysis.

10.9.1 The samples must be analyzed on a GC or GC/MS system meeting the initial calibration, continuing calibration and blank technical acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15).

10.9.2 The sample must be analyzed within the required holding time.

10.9.3 The sample must have an associated method blank meeting the blank technical acceptance criteria.

10.9.4 The percent recovery of each of the system monitoring compounds in the sample must be within the acceptance windows.

10.9.5 After analyzing a sample that exceeds the initial calibration range the analyst must either analyze an instrument blank (using the same purge inlet if using an auto sampler) which must meet technical acceptance criteria for blank analysis or monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample that exceeded the calibration range.

10.10 Corrective Action for Sample Analysis

10.10.1 Samples must meet technical acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15) before reporting data.

10.10.2 Corrective action must be completed prior to sample analysis if analysis failed to meet instrument performance checks, initial calibration, continuing calibration and method blanks.

10.10.3 Corrective action for system monitoring compounds and internal standard compounds that fail to meet acceptance criteria must be completed prior to sample analysis.

10.11 If any of the system monitoring compounds and internal standard compounds fail to meet acceptance criteria of the determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15):

10.11.1 Check all calculations, instrument logs, the system monitoring compound and internal standard compound spiking solutions and the instrument operation. If the calculations were incorrect, correct calculations and verify that the system monitoring compound recoveries and internal standard compound responses meet acceptance criteria

10.11.2 Check the preparation of the internal standards and system monitoring compounds for concentration and expiration.

10.11.3 Verify that the instrument is operating correctly.

10.12 Data that fails to meet minimum acceptance criteria will be annotated (flagged) with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a *Non-Conformance Memo* (NCM) (TestAmerica Edison Work Instruction No. EDS-WI-012) will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *Standard Operating Procedure for Control of Non-Conformances and Corrective Action* (TestAmerica Edison SOP No. EDS-GEN-003).

11.0 Calculations / Data Reduction

11.1 For specific calculations refer to the applicable determinative methods (reference the applicable TestAmerica Edison SOP; see references in Section 15

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0 Pollution Control

13.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2 The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0 Waste Management

14.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.2 The following waste streams are generated as a result of this analysis:

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system.

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene

waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium
  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

- Returned Methanol Preservative - Methanol preserved vials are collected in satellite accumulation and then transferred to 55 gallon open top steel waste drums in the waste room. These waste drums are then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, *Method 5030C: Purge and Trap for Aqueous Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3 2003.

15.2 United States Environmental Protection Agency , *Method 5035A*: *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 0, July 2002.

15.3 United States Environmental Protection Agency, “Method 8260D, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”, Test Methods for Evaluating Solid Waste, SW846, Update VI, Revision 4, June 2018.

15.4 United States Environmental Protection Agency, *Method 8015D*: *Non-Halogenated Organics using GC-FID,* Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Revision 4, June 2003.

15.5 TestAmerica Edison SOP No. ED-GCV-008, *Gasoline Range Organics using GC-FID Method, SW846 Method 801D,* current revision.

15.6 TestAmerica Edison SOP No. ED-MSV-002, *Closed System Purge and Trap and Extraction for Volatile Organics in Soil, SW846 Method 5035A,* current revision.

15.7 TestAmerica Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021* **,** current revision.

15.8 TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.9 TestAmerica Edison SOP No. EDS-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action*, most current revision

16.0 Method Modifications

Not applicable

17.0 Attachments

Not applicable

18.0 Revision History

Revision 10, dated: 02/09/2021

- Updated to Eurofins branding;
- Updated Lab Quality Manual section references as applicable throughout
- Updated Section 15 (References) as needed.

Revision 9, dated: 06/26/2013:

- Revised title of SOP to include reference to SW846 5030C.
- Section 1.1.1 and throughout document as appropriate: added reference to SW846 5030C.
- Section 1.1.1: inserted word ‘extracts’ in reference to analysis of high concentration and soil samples by SW846 5035/5035A.
- Section 10.8.10: replaced Target reference with Chrom (newly implemented chromatography data system)
- Section 15.3: corrected method reference to ‘5035A’.
- Section 15.14: added reference to SW8465030C

Revision 8, dated: 11/27/2012:

- Updated referenced Lab Quality Manual section and appendix numbers as appropriate.

Revision 7, dated: 10/02/2008

- Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).

- Section 1.2 Added reference to Quality Assurance Manual for method modifications. Added references to related methods and SOPs for determinative methods.
- Section 2 (and throughout document): added option of purging with nitrogen gas as applicable.
- Section 3: revised to reference new location for definitions.
- Section 5: Revised to include most up to date corporate health and safety references and information.
- Section 6: Newer instrumentation and GC column types added. See sections 6.1.2 and 6.1.9. All references to Tekmar instruments 2016 and 2000 and procedures to use with it removed in all relevant sections.
- Section 11: Removed reference to Organic Calculation SOP.
- Section 12: updated and revised the MDL requirements to reflect text in the current revision of the TestAmerica Edison Laboratory Quality Manual (LQM).
- References: Expanded to include more specific SOP references
- Section 18: Added this Revision History section

eurofins	Always check on-line for validity.	Level:
Document number: **T-PFAS-WI49209**	**Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633**	**Work Instruction**
Old Reference:		
Version: **5**		Organisation level: **5-Sub-BU**
Approved by: **ULJC** Effective Date: **07-MAR-2024**	Document users: **5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep**	Responsible: **5_EUUSLA_PFAS_Manager**

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Revision Log
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Revision Log

	Revision: 05	**Effective date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Scope	Clarification	Clarified carbon clean up happens during the SPE
Apparatus and Equipment	Procedure change	Added 125mL bottles. Added Sciex 7500 info. Added new pre-column.
Reagents and Standards	Clarification	Renamed attachments
Procedure	Enhancements	Replaced "500mL" with "125mL". pH is now adjusted to Proprietary Carbon cleanup is now performed during SPE.
Attachments	Clarification	Updated with new standard preps

	Revision: 04	**Effective date:** This version
Section	**Justification**	**Changes**

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	Revision: 04	**Effective date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout document	Requirement	Added references to Draft 4. Removed references to Sciex 4500.
Personnel Training and Qualification	Enhancement	Corrected table and version references
Sample Collection, Preservation, and Handling	Enhancement	added statement regarding landfill leachates
Apparatus and Equipment	Clarification	Updated to correct Cat#s, Corrected WAX cartridge
Reagents and Standards	Clarification	Updated to correct Cat#s
Reagents and Standards	Method Requirement	Corrected % purity
Calibration A	Method Requirement	Added S/N for compounds without confirmation ions Removed verbiage regarding Linear/Branched standard
Calibration A	Clarification	Removed WV requirements
Procedure B/D	Clarification	Corrected the Envi-carb and filtration steps done in in the instrument room. Corrected IS name.
Procedure E	Method Requirement	Instrument Blanks report to MDL
Procedure E	Enhancement	Added reporting procedure for samples with failing EIS and NIS. Updated RT criteria.
Procedure E	Clarification	Updated NIS criteria
Procedure E	Clarification	Mass tune must be +/- 0.2 amu
Statistical Information/Method Performance	Clarification	Updated NIS criteria
Attachment 3	Enhancement	Updated to reflect current method
Attachment 5-20	Enhancement	updated attachments to reflect new standards

Reference

1. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, 4th DRAFT, July 2023.

2. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, 3rd DRAFT, December 2022.

3. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, 2nd DRAFT, June 2022.

4. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, DRAFT, August 2021.

5. Chemical Hygiene Plan, current version.

Cross Reference

Document	Document Title
T-PFAS-WI21568	Environmental Monitoring in the PFAS Lab
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses

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Document	Document Title
T-WC-WI53304	Total Suspended Solids (TSS Gravimetric) Prescreen by EPA Draft Methods in Aqueous Samples
QA-SOP11178	Demonstrations of Capability
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
G-DC-FRM23907	Redacted SOPs

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Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in aqueous samples to include non-potable waters and non-regulatory potable water when directed by the client. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS. Compounds other than those listed may be analyzed by client request.

Analyte	Acronym	CAS#
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnDA	2058-94-8
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
8:2 - Fluorotelomersulfonic acid	8:2FTS	39108-34-4
N-methylperfluoro-1-octanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethylperfluoro-1-octanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
4:2-Fluorotelomersulfonic acid	4:2-FTS	757124-72-4
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
6:2-Fluorotelomersulfonic acid	6:2-FTS	27619-97-2
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoDS	79780-39-5
Perfluorooctanesulfonamide	PFOSA	754-91-6

Analyte	Acronym	CAS#
2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	NMePFOSAE	24448-09-7
N-methylperfluoro-1-octanesulfonamide	NMePFOSA	31506-32-8
2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	NEtPFOSAE	1691-99-2
N-ethylperfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid; (Hexafluoropropylene oxide dimer acid)	HFPODA	13252-13-6
Ammonium 4,8-dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS, F53B major	756426-58-1 *
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS, F53B minor	763051-92-9 *
3-Perfluoropropylpropanoic acid	3:3 FTCA	356-02-5
3-Perfluoropentylpropanoic acid	5:3 FTCA	914637-49-3
3-Perfluoroheptylpropanoic acid	7:3 FTCA	812-70-4
Perfluoro-3-methoxypropanoic acid	PFMPA, PFECA F	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA, PFECA A	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA, PFECA B	151772-58-6
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA, PES	113507-82-7

*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

Basic Principles

An aqueous sample is fortified with isotopically-labeled extraction standards and is passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. Carbon cleanup is performed during SPE. The extract is filtered and fortified with Isotopically-labeled injection internal standards. It is then analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where possible.

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2. Proprietary Content

3. All part of the SPE manifold must be cleaned as per *T-PFAS-WI21568*.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. IDOC trials are spiked at the OPR Level. IDOC must meet the requirements for the IPR as listed in Tables 5 and 6 of method 1633 draft version 4. See *Attachment 21*.

See *QA-SOP11178* for additional information on IDOC and DOC.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 125-mL HDPE containers. All sample containers must have linerless HDPE or polypropylene caps. Keep the sample sealed from time of collection until extraction. The entire aqueous sample that is collected must be prepared and analyzed, and subsampling must be avoided whenever possible.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

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B. Sample Storage and Shipment

1. Samples must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when the samples are received at the laboratory.

2. Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, and protected from light until extraction. Alternatively, to meet project requirements, samples may be stored at less than or equal to -20°C and protected from light until extraction.

3. Water samples must be extracted within 28 days when stored at a temperature of 0° to 6°C, not frozen, and protected from light. Water samples must be extracted within 90 days when stored at a temperature less than or equal to -20°C and protected from light. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C and protected from light. Extracts that are stored frozen and protected by light must be analyzed within 90 days.

Apparatus and Equipment

A. Apparatus

1. 125-mL HDPE bottles, CapSure Leak-Proof bottles. Environmental Sampling Supply, Catalog No. 0125-1902-QC or equivalent.

2. Centrifuge tubes, 15-mL conical polypropylene with polypropylene screw caps. Fisher Scientific, Catalog No. 05-539-5 or equivalent.

3. Volumetric flasks, 10-mL, polypropylene, Class A. Fisher Scientific, Catalog No. S02288 or equivalent.

4. HDPE bottles for extraction fluid storage, 1-L. Environmental Sampling Supply, Catalog No. 1000-1902-PC or equivalent.

5. Analytical Balance – Capable of weighing to 0.0001 g

6. Top-Loading Balance – Capable of weighing to 0.01 g

7. Solid phase extraction (SPE) Weak Anion Exchange ("WAX") cartridge, Strata-X-AW 33 µm Polymeric Weak Anion, 200 mg / 6 mL, Tubes. Phenomenex, Catalog No. 8B-S038-FCH or equivalent.

8. Large-volume SPE Reservoir (25-mL) - Millipore-Sigma, Product No. 54258-U.

9. SPE Tube Adapter - Millipore-Sigma, Catalog No. 57020-U.

10. SPE vacuum extraction manifold –"Resprep" 24-port manifold. Restek, Catalog No. 26080 or equivalent.

11. Polypropylene SPE delivery needles. Agilent, Catalog No. 12234511 or equivalent.

12. Centrifuge, "Q-Sep 3000", capable of a minimum rotational speed of 3000 rpm. Restek Corp., Catalog No. 26230 or equivalent.

13. Auto Pipettes – Eppendorf, Repeater; capable of accurately dispensing 10- to 1000-µL. Fisher Scientific Catalog No. 14-287-150 or equivalent.

14. Polypropylene pipette tips, 0.1-200 µL. Fisher Scientific, Catalog No. 02-681-135 or equivalent.

15. Polypropylene pipette tips, 100-1000 µL. Fisher Scientific, Catalog No. 02-707-508 or equivalent.

16. Polypropylene pipette tips, 1 – 10 mL. Eppendorf, Catalog No. 022492098 or equivalent.

17. Pipettes, disposable transfer. Fisher Scientific, Catalog No. 13-711-7M or equivalent.

18. Vortex mixer, variable speed. Fisher Scientific or equivalent.

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19. N-Evap sample extract concentrator with N_2 supply and water bath for temperature control. Organomation, Inc. Catalog No. 11250 or equivalent.

20. Reagent Water Purification System: Capable of producing ultrapure "Type 1/Milli-Q"-grade water from in-house deionized water system. Millipore SAS; Catalog No. FTPF08831.

21. SureStart Polyspring inserts, Catalog No. 03452340 or equivalent.

22. Short-Cap Vial with Grad Marking Spot, 9-425 Screw-Thread, 2.0 mL, 9 mm, Clear, 1000-pk., Restek, Catalog No. 21141 or equivalent.

23. Short Screw Caps, Polypropylene, Screw-Thread, PTFE/Silicone Septa, Blue, Preassembled, 2.0 mL, 9 mm, 100-pk. Restek, Catalog No. 24485 or equivalent.

24. Centrifuge tubes, 50-mL conical polypropylene with polypropylene screw caps. Fisher Scientific, Catalog No. 06-443-21 or equivalent.

25. Polypropylene bottles for standard storage, 4-mL. Fisher Scientific, Catalog No. 02-923-11E or equivalent.

26. Stainless steel spatula/scoop set. Bel-Art SP Scienceware, Product No. 11-865-130 or equivalent.

27. pH paper, Hydrion, range 5.5-8.0. Fisher Scientific, Catalog No. 14-853-275 or equivalent.

28. Syringe filter - Acrodisc, Syringe Filter, GHP,13 mm, 0.2 µm, Aqueous, 100/pkg, Part No. WAT097962 or equivalent.

29. Silanized glass wool. Sigma-Alrich, Catalog No. 20411 or equivalent.

30. Disposable syringe filter, 25-mm, 0.2um Nylon membrane, PALL/Acrodisc or equivalent.

31. Glass fiber filter, 47 mm, 1 um, PALL A/E or equivalent.

32. Wheaton Bottle, 15-mL, narrow mouth, HDPE, Leak resistant; DWK Life Sciences, Catalog No. 209044SP or equivalent.

33. Graduated Medicine Cups, Plastic, 30mL. Medline Industries Catalog No. DYND8000 or equivalent.

B. Equipment

1. AB Sciex Triple Quad 5500/5500 Plus Turbo V Ion Source or equivalent

   ExionLC Controller
   ExionLC AC Pump
   ExionLC AC Autosampler
   Exion AC Column Oven
   Data system – Analyst 1.6.3

2. AB Sciex Triple Quad 7500 QTRAP Ready or equivalent.

   Shimadzu DGU-405 Degasser
   Shimadzu LC-40D XR Pump A
   Shimadzu LC-40D XR Pump B
   Shimadzu SIL-40C XR Autosampler
   Shimadzu CBM-40 Controller
   Shimadzu CTO-40C Column Oven
   Data system – Sciex OS (Version 3.1.6.44)

3. Proprietary Content

4. HPLC columns

   a. Proprietary Content

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Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

A. Reagents

1. Methanol (MeOH) – VWR "Chromasolv LC-MS" grade Cat. No. BJ34966-4L or equivalent
2. Acetonitrile (ACN) – Fisher Scientific, Optima Cat. No. A955-4 or equivalent
3. Ammonium acetate – Sigma Aldrich, Cat. No. 431311-250G or equivalent. Store at 2-8°C.
4. Ammonium hydroxide, 30% in water, certified ACS+ grade or equivalent, store at room temperature
5. Aqueous ammonium hydroxide (3%) – add ammonium hydroxide (10 mL, 30%) to reagent water (90 mL). Store at room temperature. Replace after 3 months.
6. Methanolic ammonium hydroxide (1%) - add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month
7. Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid - add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month.
8. Acetic Acid – ACS grade. Store at room temperature. Sigma Aldrich Cat. No. 695092-100mL or equivalent.
9. Acetic Acid (0.1%) – dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.
10. Formic Acid
    a. Formic acid (aqueous, 0.1 M) - dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years
    b. Formic acid (aqueous, 0.3 M) - dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years
    c. Formic acid (aqueous, 5% v/v) - mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years
    d. Formic acid (aqueous, 50% v/v) - mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years
    e. Formic acid (methanolic 1:1, 0.1 M formic acid/methanol) - mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years
11. "Superclean Envi-Carb"; bulk sorbent. Supelco Cat# 57210-U or equivalent.
12. 20 mM ammonium acetate solution in 95:5(v/v) Milli-Q water/acetonitrile-Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 1900 mL Milli-Q water and mix well to dissolve the ammonium acetate. Add 100 mL acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.
13. 100% acetonitrile into a 2-L glass mobile phase bottle. Store at room temperature for up to 2 months. Different volumes can be prepared.

B. Standards

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Standards are prepared using calibrated pipettes, polypropylene microcentrifuge tubes, polypropylene bottles, and 10-mL Class A PP volumetric flasks to create solutions at desired concentrations. The concentrated solution is injected below the surface of the diluting solvent After preparation is completed, standards should be vortexed to ensure complete mixing. Measurement of volumes less than 5 µl should be avoided in routine production operations.

All stock, intermediate and spiking solutions are prepared using Methanol.

All initial calibration, initial calibration verification, and linear branched working standard solutions are prepared using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid.

All diluted solutions must be stored in HDPE containers that have been thoroughly rinsed with methanol.

Stock standard and intermediate standard solutions are stored in the refrigerator in labeled polypropylene screw-top vials, PP bottles, or PP centrifuge tubes.

Expiration dates are managed through LIMS Reagent. Solutions transferred from sealed glass ampules to screw-capped vials are given expiration dates of 1 year from the date opened or the expiration date provided by the vendor, whichever occurs sooner. Intermediate solutions are given an expiration date of 6 months from the preparation date, or the expiration date from the ampule provided by the vendor, whichever occurs sooner. The ampules and transferred solutions are stored in the refrigerator.

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator. When these solutions are prepared they must be tested prior to use in the PFAS extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

Working initial calibration solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working initial calibration solution, whichever occurs sooner.

The primary/preferred standard vendor is Wellington Laboratories, Inc. Ontario, Canada. Listed catalog numbers are taken from Wellington product lists. Equivalent standards may be substituted, if the listed standards are unavailable.

The solution concentration listed is as presented on the certificate of analysis and includes adjustment for purity and the salt form of the compound used.

The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See *Attachment 4*. If the compound purity is assayed to be 98% or greater, weight can be used without correction to calculate concentrations.

Log purchased standards into LIMS Reagent. Select the solution category SOURCE for purchased mixes and/or single-compound ampules. LIMS Reagent system will assign formatted names to the purchased standard solutions. The automatically-generated name can be overwritten with a manually created name if desired. Use labels printed through the LIMS Reagent to identify and track standard solutions after transfer from original ampule to storage vial. The CofA for the ampulated stock standard is attached in LIMS Reagent for reference.

Standards are prepared by transferring a known quantity of Standard to a final volume of solvent. Standard Preparation is documented in LIMS Reagent. Solutions are stored by Type in LIMS Reagent, i.e., INTERMEDIATE=working solutions and intermediate standards and SOURCE=stocks (ampulated solutions). Each Standard is given a unique name.

The following attachments provide examples of standard preparation and purchasing information. Refer to the documentation in LIMS Reagent for standards preparation information.
Attachment 5 - 1633 Native PFAS Intermediate A
Attachment 6 - 1633 Native PFAS Intermediate B
Attachment 7 - 1633 Extracted Internal Standard Intermediate
Attachment 8 - 1633 Non-Extracted Internal Standard Intermediate
Attachment 9 - 1633 TOP Surrogate Intermediate

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Attachment 10 - 1633 ICAL Standard Preparation Scheme
Attachment 11 - 1633 Initial Calibration Standard Concentrations
Attachment 12 - Bile Salts Stock Solution
Attachment 13 - Bile Salts Intermediate
Attachment 14 - Bile Salts Working Solution
Attachment 15 - 1633 WDM Standard
Attachment 16 - 1633 Native Mid-Level Spike
Attachment 17 - 1633 Low-Level Spike
Attachment 18 - 1633 1633 ICV Working Standard
Attachment 19 - 1633 Purchased/Ampulated Standards

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Calibration

A. Initial Calibration

1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order curve fit (quadratic). In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, NEtFOSAA, PFNA, PFOA, PFOSA, NEtFOSE, NMeFOSE, NEtFOSA, and NMeFOSA. S/N ratio for the lowest standard within the quantitation range must be greater than or equal to 3:1 for quantification ions and the confirmation ions, or greater than or equal to 10:1 if the analyte only has a quantification ion.

2. Analyze a Cal4 level standard that contains Bile Salt retention time markers. The analysis of this standard is used to evaluate the interference from bile salts in samples.

Example Initial Calibration Sequence:
1. Instrument Blank
2. Instrument Blank
3. Instrument Blank
4. CAL 1
5. CAL 2
6. CAL 3
7. CAL 4
8. CAL 5
9. CAL 6
10. CAL 7
11. ICB (Instrument Blank)
12. ICV
13. WDM (Bile Salts standard)

3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See *Attachment 2* for additional information about compound relationships.

4. Analyze a standard containing 100 ppb of Taurodeoxycholic Acid (TDCA), Taurochenodeoxycholic Acid (TCDCA), and Tauroursodeoxycholic Acid (TUDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in samples. The analytical conditions must be set to allow a separation of at least 1 minute between TDCA and PFOS.

Note: For better accuracy, PFTrDA is quantitated using the average of the areas of labeled compounds 13C2-PFTeDA and 13C2-PFDoDA.

Compound	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	

Compound	Extraction Standard
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

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5. Fit the curve.

   a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.

   b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%.

   c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within ±30% of its true value.

   d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

   NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See *Attachment 4*.

6. Initial Calibration Verification (ICV)

   A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock by a second analyst may be used. The calculated amount for each analyte must be within ±30% of the true value. If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.

2. Acceptance criteria

   a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

   b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

If samples are submitted in 500-mL bottles, the procedure in *Attachment 20* is followed instead.

All water samples should be evaluated for Total Suspended Solids (TSS) as per *T-WC-WI53304*. Although the full container volume of sample should be extracted, this method is applicable to aqueous samples containing up to 100 mg/L of suspended solids. For samples containing > 100mg/l of suspended solids, or when unavoidable due to high levels of PFAS, smaller samples volumes may be analyzed. However, ultimately, the TSS results, visual inspection, and analyst experience are all used to determine the volume to be used to extract the sample.

A. Sample Preparation

1. Weigh sample container with contents on a calibrated top loading balance, and record the first reading in the automated prep entry system.

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a. For all samples, the full bottle must be extracted. The sample must remain in the original sample container until after spiking solutions have been added.

b. If limited sample is submitted, spike sample in original container, then add Milli-Q water to bring to final volume of 125 mL prior to SPE extraction (see B.6 for spiking details).

c. If the sample has dissolved and/or settleable solids content (i.e; is cloudy or has a layer of sediment/solids at the bottom of the bottle), the sample must be centrifuged in order to minimize the difficulty of passing through the SPE sorbent bed. In order to preserve the integrity of the sample and ensure the full volume of the container is used, see Procedure C.

2. Use a 125 mL HDPE bottle for the method blank, the laboratory control sample (LCS) and the low level laboratory control sample (LLCS). Fill each bottle with 125 mL of Milli-Q water. Record 125 mL as the volume for the batch QC samples on the batchlog.

3. Check that the pH is proprietary content. If necessary, adjust the pH with 50% formic acid or ammonium hydroxide (or with 5% formic acid and 3% aqueous ammonium hydroxide). **NOTE: Never put pH test strips into the sample. Use a disposable pipette to place a few drops of sample onto the pH paper.**

B. Solid Phase Extraction (SPE) - Manual Sample Extraction

1. Add 10 mg of dispersive carbon to the cartridge barrel.

2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port number for each sample/QC piece on the batchlog.

3. Condition each SPE cartridge with the following reagents in the following order without allowing the cartridges to go dry:

a. 15 mL 1% methanolic ammonium hydroxide

b. 5 mL 0.3M formic acid

c. Discard conditioning eluent(s)

4. Label each sample bottle, cap and reservoir with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).

5. Vortex all spike solutions prior to use.

6. Spike QC and all samples with 25 µL of Working labeled extraction standard spike solution (1633_4X_SS_#####). Spike LCS/MS/MSD with 50 µL of native 1633 mid-level spike solution (1633_4X_MS_M_#####). Spike LLCS with 40 µL of native 1633 low-level spike solution (1633__4X_MS_L_#####). Vortex/Shake containers to mix thoroughly.

7. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges.

8. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by 5 mL 1:1 0.1M formic acid/methanol and pass the rinses through the cartridge using vacuum. Apply full vacuum (not exceeding 20" Hg) to the cartridges and dry until they are visually similar to an unused cartridge. This may take up to 15 minutes for some cartridges. Discard the rinse solution.

NOTE: If a given sample is too problematic to extract using one cartridge with full vacuum, a second cartridge is created following Procedure B.1-3 above.

9. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge ensuring the delivery needles to do not touch the sides of the tubes.

10. Rinse the inside of each empty sample/QC bottle with 5mL of 1% methanolic ammonium hydroxide. Add to reservoir and cartridge.

11. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.

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12. Disconnect the cartridge/adapter from the manifold. Remove the collection tubes.

    Note: If a second cartridge was required, the elutant from the second cartridge is added to the first cartridge elutant with a methanol rinse and then blown down to 5 mL on an N-evap prior to step 13 below.

13. Proprietary Content to each collection tube and vortex to mix.

14. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system. The prep entry system will calculate the sample weight. Record the calculated weight as the sample volume on the batchlog.

    Note: The instrument lab chemist performs the next steps.

15. Bring each sample extract to final volume 5 mL using methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

16. Add 25 µL of Internal Standard Spike Solution (1633_4X_IS_#####) to a clean 15-mL polypropylene centrifuge collection tube.

17. Cap and vortex to mix.

18. Transfer a portion of the final extract to the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

19. Cap the centrifuge tube. The remaining centrifuged extracts are stored in the refrigerator for dilution or reinjection if needed.

C. Samples containing Dissolved and/or Settleable Solids

1. Check that the pH is 6.0 ± 0.5. If necessary, adjust the pH with 50% formic acid or ammonium hydroxide (or with 5% formic acid and 3% aqueous ammonium hydroxide). **NOTE: Never put pH test strips into the sample. Use a disposable pipette to place a few drops of sample onto the pH paper.**

2. Spike sample with appropriate spikes as in Procedure B.6.

3. Centrifuge the full bottle.

4. DO NOT SHAKE BOTTLE FOLLOWING THE CENTRIFUGE STEP.

5. Follow steps in Procedure B.1 through 4.

6. Attach a 25-mL SPE reservoir to each cartridge. Decant the centrifuged sample onto its respective SPE cartridge. Allow the full volume to pass through each cartridge by gravity if possible. Apply a light vacuum if necessary. The drip rate should be approximately 1-2 drops per second.

7. Rinse the sample bottle with 5 mL of Milli-Q water, add the rinseate to the cartridge, and repeat.

8. Continue extraction process with Procedure B.8.

D. Solid Phase Extraction (SPE) – Proprietary Content

1. Proprietary Content

2. Proprietary Content

3. Proprietary Content

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4. Proprietary Content
5. Proprietary Content
6. Proprietary Content
7. Proprietary Content
8. Proprietary Content
9. Proprietary Content
10. Proprietary Content
11. Proprietary Content
12. Proprietary Content
13. Proprietary Content
14. Proprietary Content
15. Proprietary Content
16. Proprietary Content

E. LC/MS/MS Analysis

1. Mass Calibration and Tuning

    a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

    b. When masses fall outside of the ±0.2 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.2 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.

2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (5500/5500 Plus/7500) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

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3. Example acquisition method: See *Attachment 3*. Mass Transitions: See *Attachment 1*.

4. Instrument Sensitivity Check (ISC) and Instrument Blanks

    a. Prior to sample analysis, an Instrument Sensitivity Check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within ±30% of their true values. The signal-to-noise ratio must be greater than or equal to 3:1 for the quantitation ions and confirmation ions, and 10:1 for quantitation ions that have no confirmation ion. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.

    b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily at the start of a sequence. The concentration of all analytes must be less than the MDL. If acceptance criteria are not met, the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.

5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.

6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, WDM standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes Bile Salt RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

Example Sample Sequence:

1. Instrument blank
2. Instrument blank
3. Instrument blank
4. Instrument Sensitivity Check (CCV0 _CAL1)
5. CCV 2_CAL4
6. WDM (Bile Salts marker)
7. Instrument Blank (ICB)
8. Method Blank (MB)
9. Low Level LCS (LLCS)
10. LCS
11. Sample (10 or less)
12. CCV 3_CAL4
13. Instrument Blank

7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extracted method blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted. If there is no target analyte greater than the reporting limit in samples associated with an unacceptable method blank, the data may be reported with qualifiers.

9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.

    a. LLCS, LCS, MS, extraction standard recoveries, and RPDs are calculated and compared to the limits stored on the LIMS.

    b. If LLCS and LCSD recoveries are acceptable, proceed to sample quantitation.

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c. If the LCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, consult supervisor for course of action based on batch and samples results.

d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

10. Calculate the relative percent difference (RPD) between the sample and the matrix duplicate (DUP). The RPD must be less than or equal to 30%. This criteria only applies to analytes whose concentration in the sample is greater than or equal to the LOQ. If the criteria is not met, narrate with an NCM and report the data.

11. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, MS/MSD, and sample duplicates prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria defined in Table 6 of EPA 1633 Draft 4. Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-noise ratio is greater than 10:1.

12. If the extraction standard recovery(ies) is(are) outside the QC limit(s), dilute the sample extract and re-analyze. If the EIS recovery(ies) of the diluted sample extract are below 5%, a reduced sample aliquot will be extracted. If the EIS recovery(ies) of the diluted sample extract are greater than 5%, the data is reported.

13. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 50-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 50-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.

14. Compare the retention times of all of the analytes, surrogates, and internal standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than 0.4 minutes. Furthermore, native compounds with an exact isotopically-labeled analogue must elute within 0.1 minutes from their associated analogue.

15. If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.

16. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of PFBA, PFECA F(PFMPA), PFECA A(PFMBA), PFPeA, NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required.

17. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of internal standard spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%.Extracts requiring dilutions greater than 10X should be re-extracted using a reduced aliquot.

Dilution Example 1/10: Mix 895 µL of Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution with 100 µl of sample extract and 5 µl of internal standard spike solution. Vortex to mix. Using an auto-pipette, transfer an aliquot of the mixed solution into a labeled auto-sampler vial. Cap and vortex thoroughly to mix.

Calculations

1. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

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On-column Concentration = peak area ratio ÷ AVE RRF

3. On-Column Analyte Concentration using linear curve

   On-column Concentration = (peak area ratio - intercept) ÷ slope

4. Sample Concentration

   Sample concentration (ng/l) = (On-column concentration x Final Sample Volume x DF) ÷ Initial Sample Volume

5. Ion Ratio

   ion ratio = (peak area or height of quantifier)/(peak area or height of qualifier)

6. See *T-PEST-WI9847* for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

The LCS (OPR) and LLCS (LLOPR) should contain all compounds of interest. LCS, LLCS, MS, and extraction standard recoveries are compared to the limits stored on the LIMS. These limits are method defined. The LCS (OPR) and LLCS (LLOPR) and their respective EIS limits are from Table 5 and Table 6 of Draft 1633, V4. See *Attachment 21*. The MS/MSD native recoveries are compared to the OPR limits listed in Table 5 Draft 1633, V4

QC parameter	Lower acceptance limit	High acceptance limit
Non-extracted Internal Standard (NIS)	50% of the average NIS from the initial calibration	200%
Sample/Duplicate	NA	less than or equal to 30%

Historical data for MS/Ds, LCSs, EIS is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to *QA-SOP11892* for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank, an LCS/LLCS (Milli Q water spiked with all compounds to be determined carried through the entire procedure), and a sample/DUP, must be extracted and analyzed. MS/MSD is extracted only if submitted by the client. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Attachment:
Attachment 01 - Mass Transitions (.pdf)
Attachment 02 - Standard Relationships (.pdf)
Attachment 03 - Acquisition Method (.pdf)
Attachment 04 - Example Certificate of Analysis (.pdf)
Attachment 05 - Native PFAS Intermediate A (.pdf)
Attachment 06 - Native PFAS Intermediate B (.pdf)

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Attachment 07 - Extracted Internal Standard Intermediate (.pdf)
Attachment 08 - Non-extracted Internal Standard Intermediate (.pdf)
Attachment 09 - TOP Surrogate Intermediate (.pdf)
Attachment 10 - 1633 Initial Calibration Standards Preparation (.pdf)
Attachment 11 - 1633 ICAL Concentrations (.pdf)
Attachment 12 - 1633 Bile Salt Stock (.pdf)
Attachment 13 - 1633 Bile Salt Intermediate (.pdf)
Attachment 14 - 1633 Bile Salts Working Solution (.pdf)
Attachment 15 - Window-Defining Matrix Standard (.pdf)
Attachment 16 - Native Mid-Level Spike (.pdf)
Attachment 17 - Native Low-Level Spike (.pdf)
Attachment 18 - 1633 ICV Working Standard (.pdf)
Attachment 19 - Purchased Standards (.pdf)
Attachment 20 - T-PFAS-WI49209 Ver 4 (.pdf)
Attachment 21 - Tables 5, 6 (1633 Draft, V4) - Revision 4 (.pdf)

G-DC-FRM23907 Redacted SOPs
QA-SOP11178 Demonstrations of Capability
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
T-PEST-WI9847 Common Equations Used During Chromatographic Analyses
T-PFAS-WI21568 Environmental Monitoring in the PFAS Lab
T-WC-WI53304 Total Suspended Solids (TSS Gravimetric) Prescreen for PFAS by EPA Draft Methods in Aqueous Samples
Attachment: Attachment 01 - Mass Transitions (pdf)
Attachment: Attachment 02 - Standard Relationships (pdf)
Attachment: Attachment 03 - Acquisition Method (pdf)
Attachment: Attachment 04 - Example Certificate of Analysis (pdf)
Attachment: Attachment 05 - Native PFAS Intermediate A (pdf)
Attachment: Attachment 06 - Native PFAS Intermediate B (pdf)
Attachment: Attachment 07 - Extracted Internal Standard Intermediate (pdf)
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Attachment: Attachment 17 - Native Low-Level Spike (pdf)
Attachment: Attachment 18 - 1633 ICV Working Standard (pdf)
Attachment: Attachment 19 - Purchased Standards (pdf)
Attachment: Attachment 20 - T-PFAS-WI49209 Ver 4 (pdf)
Attachment: Attachment 21 - Tables 5, 6 (1633 Draft, V4) - Revision 4 (pdf)

End of document

Version history

Version	Approval	Revision information	
3	09.MAY.2023		
4	07.DEC.2023		
5	07.MAR.2024		

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Attachment 1 – Mass Transitions for AB Sciex LCMSMS

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216.0	172.0
13C4-PFBA	216.8	171.9
PFBA	212.8	168.9
13C5-PFPeA	268.3	223
PFPeA	263.0	219.0
PFPeA (2)	263.0	68.9
13C3-PFBS	302.1	79.9
13C3-PFBS (2)	302.1	98.9
PFBS	298.7	79.9
PFBS (2)	298.7	98.8
13C2-4:2-FTS	329.1	80.9
13C2-4:2-FTS (2)	329.1	309.0
4:2-FTS	327.1	307.0
4:2-FTS (2)	327.1	80.9
13C2-PFHxA	315.1	270.0
13C2-PFHxA (2)	315.1	119.4
13C5-PFHxA	318.0	273.0
13C5-PFHxA (2)	318.0	120.3
PFHxA	313.0	269.0
PFHxA (2)	313.0	118.9
PFPeS	349.1	79.9
PFPeS (2)	349.1	98.9
18O2-PFHxS	403.0	83.9
13C3-PFHxS	402.1	79.9
13C3-PFHxS (2)	402.1	98.9
PFHxS	398.7	79.9
PFHxS (2)	398.7	98.9
13C4-PFHpA	367.1	322.0
PFHpA	363.1	319.0
PFHpA (2)	363.1	169.0
13C2-6:2-FTS	429.1	80.9
13C2-6:2-FTS (2)	429.1	409.0
6:2-FTS	427.1	407.0
6:2-FTS (2)	427.1	80.9
PFHpS	449.0	79.9
PFHpS (2)	449.0	98.8
13C4-PFOA	417.1	172.0
13C8-PFOA	421.1	376.0
PFOA	413.0	369.0
PFOA (2)	413.0	169.0

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Attachment 1 – Mass Transitions for AB Sciex LCMSMS

Compound	Parent Ion	Daughter Ion
13C4-PFOS	502.8	79.9
13C4-PFOS (2)	502.8	98.9
13C8-PFOS	507.1	79.9
13C8-PFOS (2)	507.1	98.9
PFOS	498.9	79.9
PFOS (2)	498.9	98.8
13C5-PFNA	468.0	423.0
13C9-PFNA	472.1	427.0
PFNA	463.0	419.0
PFNA (2)	463.0	219.0
13C8-PFOSA	506.1	77.8
PFOSA	498.1	77.9
PFOSA (2)	498.1	478.0
PFNS	548.8	79.9
PFNS (2)	548.8	98.8
13C2-PFDA	515.1	470.1
13C6-PFDA	519.1	474.1
PFDA	512.9	469.0
PFDA (2)	512.9	219.0
13C2-8:2-FTS	529.1	80.9
13C2-8:2-FTS (2)	529.1	509.0
8:2-FTS	527.1	507.0
8:2-FTS (2)	527.1	80.8
d7-NMePFOSAE	623.2	58.9
NMePFOSAE	616.1	58.9
d3-NMePFOSA	515.0	219.0
NMEPFOSA	511.9	219.0
NMEPFOSA (2)	511.9	169.0
d3-NMeFOSAA	573.2	419.0
NMeFOSAA	570.1	419.0
NMeFOSAA (2)	570.1	483.0
d9-NEtPFOSAE	639.2	58.9
NEtPFOSAE	630.0	58.9
d5-NETPFOSA	531.1	219.0
NEtPFOSA	526.0	219.0
NEtPFOSA (2)	526.0	169.0
PFDS	599.0	79.9
PFDS (2)	599.0	98.8
13C7-PFUnDA	570.0	525.1
PFUnDA	563.1	519.0

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Attachment 1 – Mass Transitions for AB Sciex LCMSMS

Compound	Parent Ion	Daughter Ion
PFUnDA (2)	563.1	269.1
d5-NEtFOSAA	589.2	419.0
NEtFOSAA	584.2	419.1
NEtFOSAA (2)	584.2	526.0
13C2-PFDoDA	615.1	570.0
PFDoDA	613.1	569.0
PFDoDA (2)	613.1	319.0
PFDoS	699.1	79.9
PFDoS (2)	699.1	98.8
PFTrDA	663.0	619.0
PFTrDA (2)	663.0	168.9
13C2-PFTeDA	715.2	670.0
PFTeDA	713.1	669.0
PFTeDA (2)	713.1	168.9
13C3-HFPODA	286.9	168.9
13C3-HFPODA (2)	286.9	184.9
HFPODA	284.9	168.9
HFPODA (2)	284.9	184.9
DONA	376.9	250.9
DONA (2)	376.9	84.8
9Cl-PF3ONS	530.8	351.0
9Cl-PF3ONS (2)	532.8	353.0
11Cl-PF3OUdS	630.9	450.9
11Cl-PF3OUdS (2)	632.9	452.9
PFECA B (NFDHA)	295.0	201.0
PFECA B(NFDHA) (2)	295.0	84.9
PFECA F (PFMPA)	229.0	84.9
3:3 FTCA	241.0	177.0
3:3 FTCA (2)	241.0	117.0
PFECA A (PFMBA)	279.0	85.1
PFEESA (PES)	314.8	134.9
PFEESA (PES) (2)	314.8	82.9
5:3 FTCA	341.0	237.1
5:3 FTCA (2)	341.0	217.0
7:3 FTCA	441.0	316.9
7:3 FTCA (2)	441.0	336.9
FBSA	298	78
PFECHS	461	381
PFECHS (2)	461	99
FHxSA	398	78

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Attachment 1 – Mass Transitions for AB Sciex LCMSMS

Compound	Parent Ion	Daughter Ion
PFPrA	163	119
PFODA	913	869
PFODA (2)	913	169
TFSI	280	147

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Attachment 2 - PFAS Injection Standards/Extraction Standards/Native Compound

Injection Standards

Inj Std	Internal Standard/Injection Standard
I13C3-PFBA	13C3-PFBA
I13C2-PFHxA	13C2-PFHxA
I13C4-PFOA	13C4-PFOA
I13C5-PFNA	13C5-PFNA
I13C2-PFDA	13C2-PFDA
I18O2-PFHxS	18O2-PFHxS
I13C4-PFOS	13C4-PFOS

Extraction Standards

Extraction Standard	Internal Standard
E13C4-PFBA	13C3-PFBA
E13C5-PFPeA	13C2-PFHxA
E13C5-PFHxA	
E13C4-PFHpA	
E13C3-HFPO-DA	
E13C8-PFOA	13C4-PFOA
E13C9-PFNA	13C5-PFNA
E13C6-PFDA	13C2-PFDA
E13C7-PFUnA	
E13C2-PFDoA	
E13C2-PFTeDA	
E13C3-PFBS	18O2-PFHxS
E13C3-PFHxS	
E13C2-4:2-FTS	
E13C2-6:2-FTS	
E13C2-8:2-FTS	
E13C8-PFOS	13C4-PFOS
E13C8-PFOSA	
Ed3-NMeFOSA	
Ed5-NEtFOSA	
Ed3-NMeFOSAA	
Ed5-NEtFOSAA	
Ed7-NMeFOSE	
Ed9-NEtFOSE	

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Attachment 2 - PFAS Injection Standards/Extraction Standards/Native Compound

Native PFAS Compounds

Native	Extraction Standard
PFBA	13C4-PFBA
PFPrA	
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFODA	
PFBS	13C3-PFBS
FBSA	
PFPeS	13C3-PFHxS
PFHxS	
FHxSA	
PFECHS	
TFSI	13C3-PFHxS
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA

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Attachment 2 - PFAS Injection Standards/Extraction Standards/Native Compound

Native	Extraction Standard
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

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MS Method

File Info

Method File Properties

Method Name: 24JAN17-1633
Method Locked: No
Method Modified Date: 1/17/2024 4:54:04 PM
File Path: D:\SCIEX OS Data\1633\Acquisition Methods
User Name: US19_USR_INS00011E
Print Time: 1/25/2024 4:34:23 PM

Method Parameters

General

Method duration (minutes): 12.5
Target Cycle Time (ms): 400
Minimum Dwell Time (ms): 3
Maximum Dwell Time (ms): 250

Ion Source

Source name: E-ANLYT 200+ uL
Curtain gas (psi): 40
Ion source gas 1 (psi): 35
Ion source gas 2 (psi): 50
Source temperature (°C): 400

Experiment

Scan type: MRM
Polarity: Negative
Spray voltage (V): 3000
CAD gas: 9
Pause time (ms): 5
Settling time (ms): 0
Q0 dissociation: True
Q0 dissociation Simple: True
MRM Mode: Scheduled MRM

Mass Table

Group ID	Compound ID	Retention time (min)	Retention time tolerance (+/- s)	Q1 mass (Da)	Q3 mass (Da)	Custom dwell time	Dwell time (ms)	EP (V)	CE (V)	CXP (V)	Q0D (V)	Q1 resolution	Q3 resolution
PFBA	PFBA	2.74	15	213	169	False	67.3927263212977	-10	-11	-11	-20	Unit	Unit
PFPeA	PFPeA_1	3.25	20	263	219	False	17.0517944645526	-10	-12	-11	-20	Unit	Unit
PFPeA	PFPeA_2	3.25	20	263	68.9	False	17.0517944645526	-10	-55	-11	-20	Unit	Unit
PFHxA	PFHxA_1	3.56	20	313	269	False	5.89587126427121	-10	-12	-11	-20	Unit	Unit
PFHxA	PFHxA_2	3.56	20	313	118.9	False	5.89587126427121	-10	-28	-11	-20	Unit	Unit
PFHpA	PFHpA_1	3.82	20	363.1	319	False	4.32031463625942	-10	-12	-11	-20	Unit	Unit
PFHpA	PFHpA_2	3.82	20	363.1	169	False	4.32031463625942	-10	-24	-11	-20	Unit	Unit
PFOA	PFOA_1	4.06	20	413	369	False	4.02850107914667	-10	-14	-11	-20	Unit	Unit
PFOA	PFOA_2	4.07	20	413	169	False	4.06487344030903	-10	-23	-11	-20	Unit	Unit
PFNA	PFNA_1	4.3	20	463	419	False	4.45202067602072	-10	-14	-11	-20	Unit	Unit
PFNA	PFNA_2	4.3	20	463	219	False	4.45202067602072	-10	-24	-11	-20	Unit	Unit
PFDA	PFDA_1	4.57	20	512.9	469	False	5.86546025991722	-10	-16	-11	-20	Unit	Unit
PFDA	PFDA_2	4.56	20	512.9	219	False	5.77363289536258	-10	-30	-11	-20	Unit	Unit
PFUnA	PFUnA_1	4.92	20	563.1	519	False	12.126761321189	-10	-18	-11	-20	Unit	Unit
PFUnA	PFUnA_2	4.92	20	563.1	269.1	False	12.126761321189	-10	-25	-11	-20	Unit	Unit
PFDoA	PFDoA_1	5.38	20	613	569	False	26.3915641790238	-10	-18	-11	-20	Unit	Unit
PFDoA	PFDoA_2	5.38	20	613	319	False	26.3915641790238	-10	-28	-11	-20	Unit	Unit
PFTrDA	PFTrDA_1	5.84	20	663	619	False	29.9516436811519	-10	-20	-11	-20	Unit	Unit
PFTrDA	PFTrDA_2	5.83	20	663	168.9	False	29.8718690247616	-10	-36	-11	-20	Unit	Unit
PFTeDA	PFTeDA_1	6.23	20	713.1	669	False	29.0161667227783	-10	-22	-11	-20	Unit	Unit
PFTeDA	PFTeDA_2	6.23	20	713.1	168.9	False	29.0161667227783	-10	-38	-11	-20	Unit	Unit
PFBS	PFBS_1	3.66	20	298.7	79.9	False	4.93216684727113	-10	-65	-11	-20	Unit	Unit
PFBS	PFBS_2	3.66	20	298.7	98.8	False	4.93216684727113	-10	-40	-11	-20	Unit	Unit
PFPeS	PFPeS_1	3.95	25	349	79.9	False	4.22145928225894	-10	-75	-11	-20	Unit	Unit
PFPeS	PFPeS_2	3.95	25	349	98.8	False	4.22145928225894	-10	-38	-11	-20	Unit	Unit
PFHxS	PFHxS_1	4.21	25	398.7	79.9	False	4.28967109661652	-10	-80	-11	-20	Unit	Unit
PFHxS	PFHxS_2	4.21	25	398.7	98.8	False	4.28967109661652	-10	-75	-11	-20	Unit	Unit

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PFHpS	PFHpS_1	4.47	20	449	79.9	False	5.18229607918067	-10	-95	-11	-20	Unit	Unit
PFHpS	PFHpS_2	4.47	20	449	98.8	False	5.18229607918067	-10	-80	-11	-20	Unit	Unit
PFOS	PFOS_1	4.79	20	498.9	79.9	False	8.21861825334177	-10	-108	-11	-20	Unit	Unit
PFOS	PFOS_2	4.79	20	498.9	98.8	False	8.21861825334177	-10	-85	-11	-20	Unit	Unit
PFNS	PFNS_1	5.23	20	548.8	79.9	False	22.8205633748214	-10	-100	-11	-20	Unit	Unit
PFNS	PFNS_2	5.23	20	548.8	98.8	False	22.8205633748214	-10	-100	-11	-20	Unit	Unit
PFDS	PFDS_1	5.69	20	599	79.9	False	27.1837224706077	-10	-110	-11	-20	Unit	Unit
PFDS	PFDS_2	5.69	20	599	98.8	False	27.1837224706077	-10	-100	-11	-20	Unit	Unit
PFDoS	PFDoS_1	6.45	20	699.1	79.9	False	26.7393505098423	-10	-150	-11	-20	Unit	Unit
PFDoS	PFDoS_2	6.45	20	699.1	98.8	False	26.7393505098423	-10	-130	-11	-20	Unit	Unit
4-2-FTS	4-2-FTS_1	3.44	15	327.1	307	False	7.49143208479874	-10	-28	-11	-20	Unit	Unit
4-2-FTS	4-2-FTS_2	3.44	15	327.1	80.9	False	7.49143208479874	-10	-50	-11	-20	Unit	Unit
6-2-FTS	6-2-FTS_1	3.94	15	427.1	407	False	4.03834949315404	-10	-33	-11	-20	Unit	Unit
6-2-FTS	6-2-FTS_2	3.94	15	427.1	80.9	False	4.03834949315404	-10	-65	-11	-20	Unit	Unit
8-2-FTS	8-2-FTS_1	4.41	15	527.1	507	False	4.61268966639502	-10	-39	-11	-20	Unit	Unit
8-2-FTS	8-2-FTS_2	4.41	15	527.1	80.9	False	4.61268966639502	-10	-85	-11	-20	Unit	Unit
PFOSA	PFOSA_1	5.69	20	498.1	77.9	False	27.1837224706077	-10	-90	-11	-20	Unit	Unit
PFOSA	PFOSA_2	5.69	20	498.1	478	False	27.1837224706077	-10	-35	-11	-20	Unit	Unit
NMeFOSA	NMeFOSA_1	6.61	20	511.9	219	False	27.8235729055401	-10	-35	-11	-20	Unit	Unit
NMeFOSA	NMeFOSA_2	6.61	20	511.9	169	False	27.8235729055401	-10	-40	-11	-20	Unit	Unit
NEtFOSA	NEtFOSA_1	6.88	20	526	219	False	46.3426571897647	-10	-35	-11	-20	Unit	Unit
NEtFOSA	NEtFOSA_2	6.88	20	526	169	False	46.3426571897647	-10	-35	-11	-20	Unit	Unit
N-MeFOSAA	N-MeFOSAA_1	4.52	25	570	419	False	5.79679604401482	-10	-28	-11	-20	Unit	Unit
N-MeFOSAA	N-MeFOSAA_2	4.52	25	570	483	False	5.79679604401482	-10	-22	-11	-20	Unit	Unit
N-EtFOSAA	N-EtFOSAA_1	4.67	25	584	419	False	7.10779258317214	-10	-28	-11	-20	Unit	Unit
N-EtFOSAA	N-EtFOSAA_2	4.67	25	584	526	False	7.10779258317214	-10	-30	-11	-20	Unit	Unit
N-MeFOSE	N-MeFOSE	6.48	15	616.1	58.9	False	25.2584869343111	-10	-68	-11	-20	Unit	Unit
N_EtFOSE	N_EtFOSE	6.76	15	630	58.9	False	33.111343165691	-10	-66	-11	-20	Unit	Unit
HFPO-DA	HFPO-DA_1	3.67	20	284.9	168.9	False	4.88578009536273	-10	-12	-11	-20	Unit	Unit
HFPO-DA	HFPO-DA_2	3.67	20	284.9	184.9	False	4.88578009536273	-10	-18	-11	-20	Unit	Unit
ADONA	ADONA_1	3.9	15	376.9	250.9	False	4.06647300305893	-10	-40	-11	-20	Unit	Unit
ADONA	ADONA_2	3.9	15	376.9	84.8	False	4.06647300305893	-10	-48	-11	-20	Unit	Unit
9Cl-PF3ONS	9Cl-PF3ONS_1	5.12	20	530.8	351	False	20.0138934607134	-10	-38	-11	-20	Unit	Unit
9Cl-PF3ONS	9Cl-PF3ONS_2	5.12	20	532.8	353	False	20.0138934607134	-10	-38	-11	-20	Unit	Unit
11Cl-PF3OUdS	11Cl-PF3OUdS_1	6.01	20	630.9	450.9	False	30.7157068520705	-10	-41	-11	-20	Unit	Unit
11Cl-PF3OUdS	11Cl-PF3OUdS_2	6.01	20	632.9	452.9	False	30.7157068520705	-10	-41	-11	-20	Unit	Unit
3-3-FTCA	3-3-FTCA_1	3.03	20	241	177	False	31.6749443138274	-10	-12	-11	-20	Unit	Unit
3-3-FTCA	3-3-FTCA_2	3.03	20	241	117	False	31.6749443138274	-10	-50	-11	-20	Unit	Unit
5-3-FTCA	5-3-FTCA_1	3.72	20	341	237.1	False	4.69273434619782	-10	-20	-11	-20	Unit	Unit
5-3-FTCA	5-3-FTCA_2	3.72	20	341	217	False	4.69273434619782	-10	-35	-11	-20	Unit	Unit
7-3-FTCA	7-3-FTCA_1	4.26	20	441	316.9	False	4.41255274648394	-10	-30	-11	-20	Unit	Unit
7-3-FTCA	7-3-FTCA_2	4.27	20	441	336.9	False	4.41608456819758	-10	-20	-11	-20	Unit	Unit
PFEESA	PFEESA_1	3.81	20	314.8	134.9	False	4.3546637559744	-10	-35	-11	-20	Unit	Unit
PFEESA	PFEESA_2	3.81	20	314.8	82.9	False	4.3546637559744	-10	-25	-11	-20	Unit	Unit
PFMPA	PFMPA	2.97	20	229	84.9	False	38.0941171264429	-10	-25	-11	-20	Unit	Unit
PFMBA	PFMBA	3.35	20	279	84.9	False	13.0625041116259	-10	-25	-11	-20	Unit	Unit
NFDHA	NFDHA_1	3.53	20	295	201	False	6.41441552065717	-10	-15	-11	-20	Unit	Unit
NFDHA	NFDHA_2	3.53	20	295	84.9	False	6.41441552065717	-10	-40	-11	-20	Unit	Unit

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TDCA	TDCA	4.16	60	498	80	False	6.89582152082278	-10	-100	-11	-20	Unit	Unit
TDCA	TCDCA	4.17	60	498.1	80	False	6.94199635394513	-10	-100	-11	-20	Unit	Unit
TDCA	TUDCA	4.15	60	498.2	80	False	6.92164980149871	-10	-100	-11	-20	Unit	Unit
	NIS-PFBA-13C3	2.73	20	216	172	False	76.7704097249552	-10	-11	-11	-20	Unit	Unit
	NIS-PFHxA-13C2_1	3.55	20	315.1	270	False	6.04017140857135	-10	-12	-11	-20	Unit	Unit
	NIS-PFHxA-13C2_2	3.57	20	315.1	119.4	False	5.82581071987951	-10	-28	-11	-20	Unit	Unit
	NIS-PFOA-13C4	4.04	20	417.1	172	False	4.02850107914667	-10	-14	-11	-20	Unit	Unit
	NIS-PFNA-13C5	4.3	20	468	423	False	4.45202067602072	-10	-14	-11	-20	Unit	Unit
	NIS-PFDA-13C6	4.56	20	515.1	470.1	False	5.77363289536258	-10	-16	-11	-20	Unit	Unit
	NIS-PFHxS 18O2	4.21	20	403	83.9	False	4.35047589014871	-10	-74	-11	-20	Unit	Unit
	NIS-PFOS-13C4_1	4.79	20	502.8	79.9	False	8.21861825334177	-10	-85	-11	-20	Unit	Unit
	NIS-PFOS-13C4_2	4.79	20	502.8	98.9	False	8.21861825334177	-10	-108	-11	-20	Unit	Unit
	EIS-PFBA-13C4	2.73	20	216.8	171.9	False	76.7704097249552	-10	-11	-11	-20	Unit	Unit
	EIS-PFPeA-13C5	3.24	20	268	223	False	17.6858405531441	-10	-12	-11	-20	Unit	Unit
	EIS-PFHxA-13C5	3.55	20	318	273	False	6.04017140857135	-10	-12	-11	-20	Unit	Unit
	EIS-PFHxA-13C5_2	3.55	20	318	120.3	False	6.04017140857135	-10	-12	-11	-20	Unit	Unit
	EIS-PFHpA-13C4	3.82	20	367	322	False	4.32031463625942	-10	-12	-11	-20	Unit	Unit
	EIS-PFOA-13C8	4.06	20	421	376	False	4.02850107914667	-10	-14	-11	-20	Unit	Unit
	EIS-PFNA-13C9	4.29	20	472	427	False	4.43341837279547	-10	-14	-11	-20	Unit	Unit
	EIS-PFDA-13C6	4.56	20	519	474	False	5.77363289536258	-10	-16	-11	-20	Unit	Unit
	EIS-PFUnA-13C7	4.91	20	570	525	False	11.6595694664373	-10	-18	-11	-20	Unit	Unit
	EIS-PFDoA-13C2	5.38	20	615	570	False	26.3915641790238	-10	-18	-11	-20	Unit	Unit
	EIS-PFTeDA-13C2	6.22	20	715.2	670	False	29.1735850620148	-10	-22	-11	-20	Unit	Unit
	EIS-PFBS-13C3_1	3.65	20	302.1	79.9	False	5.03360405230241	-10	-65	-11	-20	Unit	Unit
	EIS-PFBS-13C3_2	3.65	20	302.1	98.9	False	5.03360405230241	-10	-40	-11	-20	Unit	Unit
	EIS-PFHxS-13C3_1	4.21	20	402	79.9	False	4.35047589014871	-10	-74	-11	-20	Unit	Unit
	EIS-PFHxS-13C3_2	4.2	20	402	98.9	False	4.3307691607502	-10	-75	-11	-20	Unit	Unit
	EIS-PFOS-13C8_1	4.78	20	507	98.9	False	8.07116400752687	-10	-85	-11	-20	Unit	Unit
	EIS-PFOS-13C8_2	4.78	20	507	79.9	False	8.07116400752687	-10	-108	-11	-20	Unit	Unit
	EIS-4-2-FTS-13C2_1	3.43	20	329	80.9	False	9.76576969168348	-10	-50	-11	-20	Unit	Unit
	EIS-4-2-FTS-13C2_2	3.43	20	329	309	False	9.76576969168348	-10	-28	-11	-20	Unit	Unit
	EIS-6-2-FTS-13C2_1	3.92	20	429	80.9	False	4.22146051180735	-10	-65	-11	-20	Unit	Unit
	EIS-6-2-FTS-13C2_2	3.92	20	429	409	False	4.22146051180735	-10	-33	-11	-20	Unit	Unit
	EIS-8-2-FTS-13C2_1	4.4	20	529	80.9	False	4.82003075017476	-10	-85	-11	-20	Unit	Unit
	EIS-8-2-FTS-												

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	13C2_2	4.4	20	529	509	False	4.82003075017476	-10	-39	-11	-20	Unit	Unit
	EIS-PFOSA_13C8	5.69	20	506.1	77.8	False	27.1837224706077	-10	-90	-11	-20	Unit	Unit
	EIS-N-MeFOSA-D3	6.6	20	515	219	False	27.5893808680694	-10	-35	-11	-20	Unit	Unit
	EIS-N-EtFOSA-D5	6.87	20	531.1	219	False	45.3890653270819	-10	-35	-11	-20	Unit	Unit
	EIS-N-MeFOSAA-D3	4.51	20	573.2	419	False	5.44053438044589	-10	-28	-11	-20	Unit	Unit
	EIS-N-EtFOSAA-D5	4.66	20	589.2	419	False	6.82400177208745	-10	-28	-11	-20	Unit	Unit
	EIS-N-MeFOSE-D7	6.46	20	623.2	58.9	False	26.7393505098423	-10	-28	-11	-20	Unit	Unit
	EIS-N-EtFOSE-D9	6.74	20	639.2	58.9	False	36.2605904961277	-10	-28	-11	-20	Unit	Unit
	EIS-HFPO-DA-13C3_1	3.67	20	286.9	168.9	False	4.88578009536273	-10	-12	-11	-20	Unit	Unit
	EIS-HFPO-DA-13C3_2	3.67	20	286.9	184.9	False	4.88578009536273	-10	-18	-11	-20	Unit	Unit
	13C3-PFPeA	3.3	20	266	222	False	14.9856787620733	-10	-14	-11	-20	Unit	Unit
	13C2-PFOA	4.05	20	415	370	False	4.02086470787393	-10	-16	-11	-20	Unit	Unit
	13C2-PFUnDA	4.91	20	565	520	False	11.6595694664373	-10	-19	-11	-20	Unit	Unit
	PFPrA	1.37	20	163	119	False	250	-10	-15	-11	-20	Unit	Unit
	PFODA	7.45	20	913	869	False	150.654269972452	-10	-25	-11	-20	Unit	Unit
	PFODA_2	7.44	20	913	169	False	146.522038567493	-10	-45	-11	-20	Unit	Unit
	TFSI	4.06	20	280	147	False	4.02850107914667	-10	-35	-11	-20	Unit	Unit
	TFSI_2	4.06	20	280	78	False	4.02850107914667	-10	-20	-11	-20	Unit	Unit
	PFECHS	4.44	20	461	381	False	5.01009906786485	-10	-40	-11	-20	Unit	Unit
	PFECHS_2	4.44	20	461	99	False	5.01009906786485	-10	-60	-11	-20	Unit	Unit
	FBSA	4.05	20	298	78	False	4.02086470787393	-10	-45	-11	-20	Unit	Unit
	FHxSA	4.74	20	398	78	False	7.71684940538199	-10	-65	-11	-20	Unit	Unit

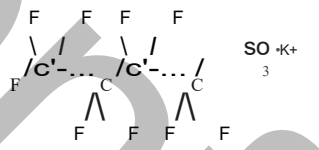
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CERTIFICATE OF ANALYSIS
DOCUMENTATION

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PRODUCT CODE: L-PFBS
LOT NUMBER: LPFBS1022
COMPOUND: Potassium perfluoro-1-butanesulfonate
STRUCTURE:
CAS #: 29420-49-3



MOLECULAR FORMULA: Cl9S03K
MOLECULAR WEIGHT: 338.19
CONCENTRATION: 50.0 ± 2.5 µg/ml (K salt)
44.4 ± 2.2 µg/mL (PFBS acid)
44.2 ± 2.2 µg/mL (PFBS anion)
SOLVENT<SI: Methanol
CHEMICAL PURITY: >98%
LAST TESTED: 1mrn1dd/yyyy) 10/04/2022
EXPIRY DATE: (mrnldd/yyyy) 10/04/2027
RECOMMENDED STORAGE: Store ampoule in a cool, dark place

DOCUMENTATION/ DATA ATTACHED:

Figure 1: LC/MS Data (Full Scan and Mass Spectrum)
Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

See page 2 for further details.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Certified By: -
B.G. Chittim, General Manager
Date: 10/14/2022
(mrn/dd/yyyy)

Wellington Laboratories Inc., 345 Southgate Dr. Guelph ON N1G 3M5 CANADA
519-822-2436 • Fax: 519-822-2849 • info@well-labs.com

Attachment "Attachment 12 - 1633 Bile Salt Stock" to "US Eurofins US Lancaster Laboratories Environmental - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633"
Printed by Erin Shortes, d. Fri 08 Mar 2024 11:27 EST

INTENDED USE:

The products prepared by Wellington Laboratories Inc. are for laboratory use only. This certified reference material (CRM) was designed to be used as a standard for the identification and/or quantification of the specific chemical compound it contains.

HANDLING:

This product should only be used by qualified personnel familiar with its potential hazards and trained in the handling of hazardous chemicals. Due care should be exercised to prevent unnecessary human contact or ingestion. All procedures should be carried out in a well-functioning fume hood and suitable gloves, eye protection, and clothing should be worn at all times. Waste should be disposed of according to national and regional regulations. Safety Data Sheets (SDSs) are available upon request.

SYNTHESIS / CHARACTERIZATION:

Our products are synthesized using single-product unambiguous routes whenever possible. They are then characterized, and their structures and purities confirmed, using a combination of the most relevant techniques, such as NMR, GC/MS, LC/MS/MS, SFC/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products are compared to older lots in the same manner, which further confirms the homogeneity of the crystalline material as well as the stability and homogeneity of the solutions in the storage containers. In order to maintain the integrity of the assigned value(s), and associated uncertainty, the dilution or injection of a subsample of this product should be performed using calibrated measuring equipment.

UNCERTAINTY:

The maximum combined relative standard uncertainty of our reference standard solutions is calculated using the following equation:

The combined relative standard uncertainty, u,(Y), of a value y and the uncertainty of the independent parameters $x_1, x_2, \ldots x_n$ on which it depends is:

$$u_c(y(x_1,x_2,\ldots x_n)) = \sqrt{\sum u(y,x_i)^2}$$

where x is expressed as a relative standard uncertainty of the individual parameter.

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of ±5% (calculated with a coverage factor of 2 and a level of confidence of 95%) is stated on the Certificate of Analysis for all of our products.

TRACEABILITY:

All reference standard solutions are traceable to specific crystalline lots. The microbalances used for solution preparation are regularly calibrated by an external ISO/IEC 17025 accredited laboratory. In addition, their calibration is verified prior to each weighing using calibrated external weights traceable to an ISO/IEC 17025 accredited laboratory. All volumetric glassware used is calibrated, of Class A tolerance, and traceable to an ISO/IEC 17025 accredited laboratory. For certain products, traceability to international interlaboratory studies has also been established.

EXPIRY DATE/ PERIOD OF VALIDITY:

Ongoing stability studies of this product have demonstrated stability in its composition and concentration, until the specified expiry date, in the unopened ampoule. Monitoring for any degradation or change in concentration of the listed analyte(s) is performed on a routine basis.

LIMITED WARRANTY:

At the time of shipment, all products are warranted to be free of defects in material and workmanship and to conform to the stated technical and purity specifications.

QUALITY MANAGEMENT:

This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAi Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A1226), and ISO 17034 by ANSI National Accreditation Board (ANAB; AR-1523).

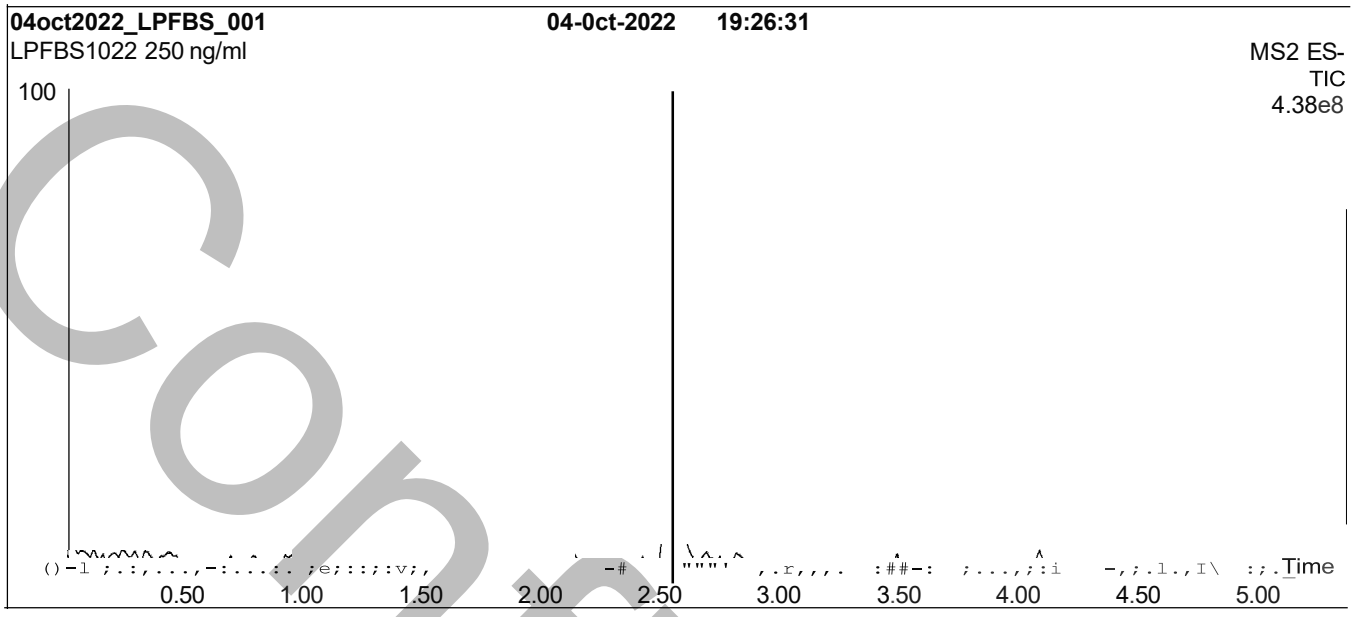
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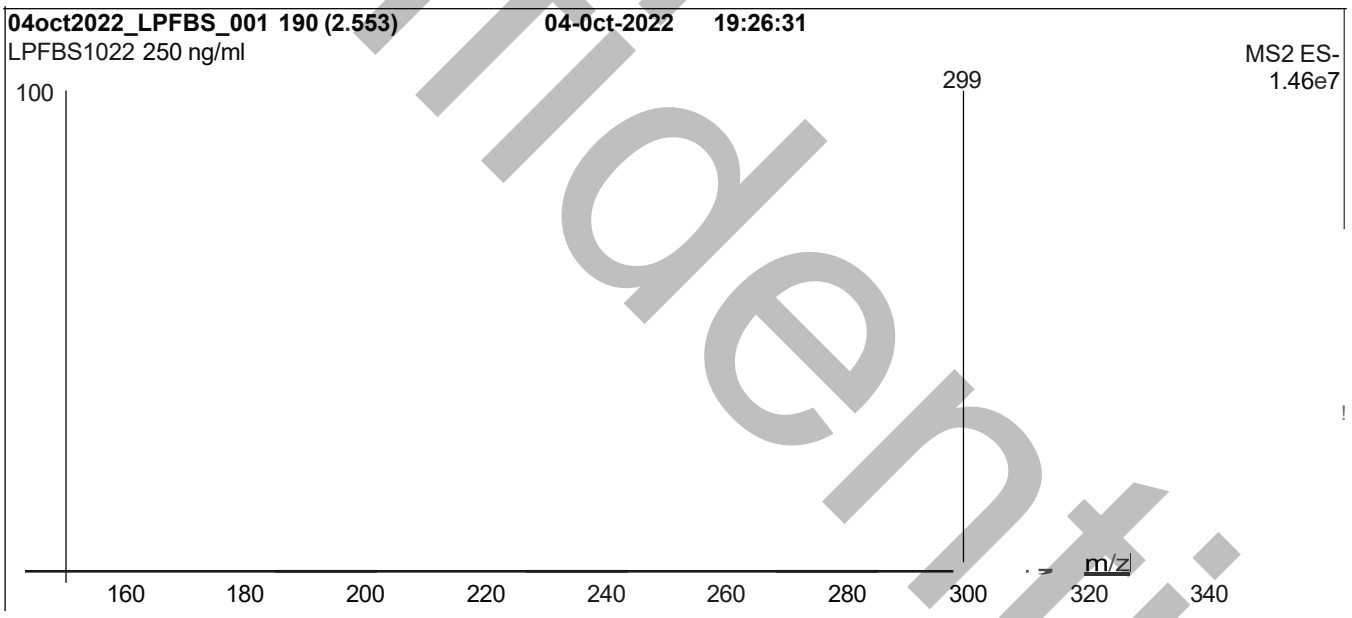
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REFERENCE MATERIAL PRODUCER

•*For additional information or assistance concerning this or any other products from Wellington Laboratories Inc., please visit our website at www.well-labs.com or contact us directly at info@well-labs.com**

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Figure 1: **L-PFBS; LC/MS Data (Full Scan and Mass Spectrum)**





Conditions **for** Figure **1:**

Waters Acquity Ultra Performance LC
Waters *Xevo* TQ-S micro MS

Chromatographic Conditions:

Column: Acquity UPLC BEH Shield RP,,
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 60% H,O / 40% (80:20 MeOH:ACN)
(both with 10 mM NH,OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions over 0.75 min.
Time: 12 min

Flow: 300 µUmin

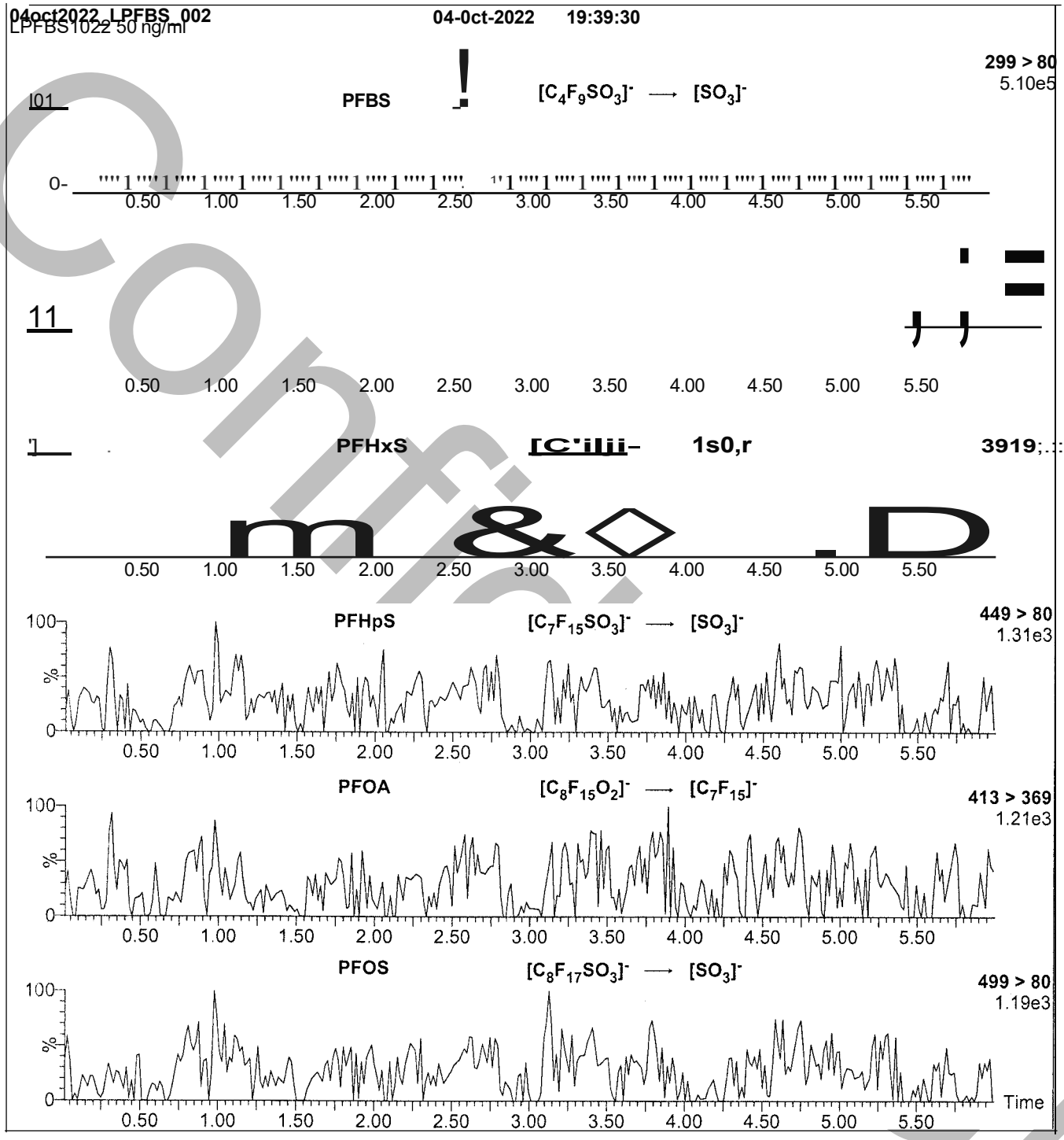
MS Parameters:

Experiment: Full Scan (150 - 850 amu)

Source: Electrospray (negative)
Capillary Voltage (kV) = 2.00
Cone Voltage (V) = 10.00
Desolvation Temperature (°C) = 500
Desolvation Gas Flow (L/hr) = 1000

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Figure 2: **L-PFBS; LC/MS/MS Data (Selected MRM Transitions)**



onditions for Figure 2:

Injection:	On-column (L-PFBS)	**MS Parameters:**	
Mobile phase:	Same as Figure 1	Collision Gas (mbar) = 3.29e-3	
Flow:	300 µUmin	Collision Energy (eV) = 30	

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Attachment 5 - Native PFAS Intermediate A								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Stock Conc. (ng/mL)	Aliquot (mL)	Final Volume of MeOH (mL)	Final Conc. Native PFAS Intermediate A (ng/mL)
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.0625		294.4
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.0625		291.3
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	47100	0.0250		117.8
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	50000	0.0187		93.5
Wellington	8;2 FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	47900	0.1000		479.0
Wellington	4;2 FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	46700	0.0625		291.9
Wellington	6;2 FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	47400	0.1000		474.0
Wellington	br-NEtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.0250		125.0
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.0250		125.0
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.0250		110.5
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.0250		125.0
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.0250		120.5
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.0250		125.0
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	48400	0.0250		121.0
Wellington	MPFDOA	Perfluorododecanoic acid	307-55-1	PFDoDA	50000	0.0250		125.0
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.0250		119.0
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.0250		125.0
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.0250		113.8
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.0250		125.0
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.0250		120.0
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.0250		125.0
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.0250		125.0
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46600	0.0250		116.5
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.0250	10	125.0
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900	0.0250		117.3
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.0250		125.0
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.0250		125.0
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.0250		125.0
Wellington	PFPUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	50000	0.0250		125.0
Wellington	PF4OPeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.0500		250.0
Wellington	PF5OHxA	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	50000	0.0500		250.0
Wellington	3,6-OPFHpA	Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50000	0.0250		125.0
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.0500		222.5
Wellington	br-NMeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSAE	50000	0.1250		625.0
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	50000	0.0250		125.0
Wellington	br-NMeFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	1691-99-2	NEtPFOSAE	50000	0.2500		1250.0
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	50000	0.0250		125.0
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.125		625
Wellington	FPePA	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	50000	0.25		1250
Wellington	FHpPA	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	50000	0.25		1250
Wellington	PFECHS	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	46100	0.025		115.25
Wellington	FBSA-I	Perfluoro-1-butanesulfonamide	30334-69-1	FBSA	50000	0.025		125
Wellington	FHxSA-I	Perfluoro-1-hexanesulfonamide	41997-13-1	FHxSA	50000	0.025		125
Wellington	PFPrA	Perfluoropropanoic acid	422-64-0	PFPrA	50000	0.025		125
Wellington	PFODA	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	50000	0.025		125
AccuStandard	PFOS-030S	Bis(trifluoromethylsulfonyl)amine	82113-65-3	TFSI	97900	0.0125		122.375

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Attachment 6 - Native PFAS Intermediate B							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Stock Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume of MeOH (mL)**	**Final Conc. Native PFAS Intermediate B (ng/mL)**
Native PFAS Intermediate A	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	294	0.10	10	2.94
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	291			2.91
	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	118			1.18
	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	94			0.94
	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	479			4.79
	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	292			2.92
	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	474			4.74
	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			1.25
	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			1.25
	Perfluorobutanesulfonic acid	375-73-5	PFBS	111			1.11
	Perfluorobutanoic acid	375-22-4	PFBA	125			1.25
	Perfluorodecanesulfonic acid	335-77-3	PFDS	121			1.21
	Perfluorodecanoic acid	335-76-2	PFDA	125			1.25
	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	121			1.21
	Perfluorododecanoic acid	307-55-1	PFDoDA	125			1.25
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			1.19
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			1.25
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	114			1.14
	Perfluorohexanoic acid	307-24-4	PFHxA	125			1.25
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			1.20
	Perfluorononanoic acid	375-95-1	PFNA	125			1.25
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			1.25
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	117			1.17
	Perfluorooctanoic acid	335-67-1	PFOA	125			1.25
	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	117			1.17
	Perfluoropentanoic acid	2706-90-3	PFPeA	125			1.25
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			1.25
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			1.25
	Perfluoroundecanoic acid	2058-94-8	PFUnDA	125			1.25
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			2.50
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			2.50
	Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	125			1.25
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	223			2.23
	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSAE	625			6.25
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	125			1.25
	2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	1691-99-2	NEtPFOSAE	1250			12.50
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	125			1.25
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			6.25
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	1250			12.50
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	1250			12.50
	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	115			1.15
	Perfluoro-1-butanesulfonamide	30334-69-1	FBSA	125			1.25
	Perfluoro-1-hexanesulfonamide	41997-13-1	FHxSA	125			1.25
	Perfluoropropanoic acid	422-64-0	PFPrA	125			1.25
	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	125			1.25
	Bis(trifluoromethylsulfonyl)amine	82113-65-3	TFSI	122			1.22

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Attachment 7 - 1633 EIS Intermediate								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Stock Conc. (ng/mL)	Aliquot (mL)	Final Volume of MeOH (mL)	Final Conc. EIS Intermediate (ng/mL)
Wellington	MPFBA	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	50000	0.1000	10	500.0
Wellington	M5PFPeA	Perfluoro-n-[13C5]pentanoic acid	STL01893	13C5-PFPeA	50000	0.0500		250.0
Wellington	M5PFHxA	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	13C5-PFHxA	50000	0.0250		125.0
Wellington	M4PFHpA	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	50000	0.0250		125.0
Wellington	M8PFOA	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	50000	0.0250		125.0
Wellington	M9PFNA	Perfluoro-n-[13C9]nonanoic acid	STL02578	13C9-PFNA	50000	0.0125		62.5
Wellington	M6PFDA	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	50000	0.0125		62.5
Wellington	M7PFUdA	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	13C7-PFUnA	50000	0.0125		62.5
Wellington	MPFDoA	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	50000	0.0125		62.5
Wellington	M2PFTeDA	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	50000	0.0125		62.5
Wellington	M3PFBS	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PFBS	46500	0.0250		116.3
Wellington	M3PFHxS	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	13C3-PFHxS	47300	0.0250		118.3
Wellington	M8PFOS	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	47800	0.0250		119.5
Wellington	M8FOSA-I	erfluoro-1-[13C8]octanesulfonamide	STL01056	13C8-PFOSA	50000	0.0250		125.0
Wellington	d3-N-MeFOSAA	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	50000	0.0500		250.0
Wellington	d5-N-EtFOSAA	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	50000	0.0500		250.0
Wellington	M2-4;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	46700	0.0500		233.5
Wellington	M2-6;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	13C2-6:2FTS	47500	0.0500		237.5
Wellington	M2-8;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	13C2-8:2FTS	47900	0.0500		239.5
Wellington	M3HFPO-DA	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	13C3-HFPO-DA	50000	0.1000		500.0
Wellington	d7-N-MeFOSE-M	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	50000	0.2500		1250.0
Wellington	d9-N-EtFOSE-M	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	50000	0.2500		1250.0
Wellington	d-N-MeFOSA-M	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	50000	0.0250		125.0
Wellington	d-N-EtFOSA-M	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	50000	0.0250		125.0

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Attachment 8 - 1633 NIS Intermediate								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Stock Conc. (ng/mL)	Aliquot (mL)	Final Volume of MeOH (mL)	Final Conc. NIS Intermediate (ng/mL)
Wellington	M3PFBA	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	50000	0.0500	10	250.0
Wellington	MPFOA	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	50000	0.0250		125.0
Wellington	MPFDA	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	50000	0.0125		62.5
Wellington	MPFOS	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	47800	0.0250		119.5
Wellington	MPFNA	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	50000	0.0125		62.5
Wellington	MPFHxA	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	50000	0.0250		125.0
Wellington	MPFHxS	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	18O2-PFHxS	47300	0.0250		118.3

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Attachment 9 - 1633 TOP SS Intermediate								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Stock Conc. (ng/mL)	Aliquot (mL)	Final Volume of MeOH (mL)	Final Conc. TOP Surrogate Intermediate (ng/mL)
Wellington	M2PFOA	Perfluoro-n-(1,2-13C2)octanoic acid	STL00623	13C2-PFOA	50000	0.0250	10	125.0
Wellington	MPFUdA	Perfluoro-n-(1,2-13C2)undecanoic acid	STL00997	13C2-PFUnA	50000	0.0125		62.5
Wellington	M3PFPeA	Perfluoro-n-(3,4,5-13C3)pentanoic acid	STL03269	13C3-PFPeA	50000	0.0500		250.0

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Attachment 10 - 1633 Initial Calibration Standards Preparation							
Solution Name	**CAL1**	**CAL2**	**CAL3**	**CAL4**	**CAL5**	**CAL6**	**CAL7**
Native PFAS Intermediate A (mL)	N/A	N/A	N/A	0.05	0.10	0.25	1.25
Native PFAS Intermediate B (mL)	0.40	1.00	2.50	N/A	N/A	N/A	N/A
1633 TOP SS Intermedaite (mL)	0.05	0.05	0.05	0.05	0.05	0.05	0.05
1633 EIS Intermediate (mL)	0.05	0.05	0.05	0.05	0.05	0.05	0.05
1633 NIS Intermediate (mL)	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Final Volume (ml) (methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid)	10	10	10	10	10	10	10

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Attachment 10 - 1633 Initial Calibration Standards Preparation							
Compound Acronym	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7
	Conc. (ng/mL)	Conc. (ng/mL)	Conc. (ng/mL)	Conc. (ng/mL)	Conc. (ng/mL)	Conc. (ng/mL)	Conc. (ng/mL)
11Cl-PF3OUdS	0.118	0.294	0.736	1.472	2.94	7.36	36.8
9Cl-PF3ONS	0.117	0.291	0.728	1.456	2.91	7.28	36.4
DONA	0.047	0.118	0.294	0.589	1.18	2.94	14.7
HFPODA	0.037	0.094	0.234	0.468	0.94	2.34	11.7
8:2-FTS	0.192	0.479	1.198	2.395	4.79	11.98	59.9
4:2-FTS	0.117	0.292	0.730	1.459	2.92	7.30	36.5
6:2-FTS	0.190	0.474	1.185	2.370	4.74	11.85	59.3
NEtFOSAA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
NMeFOSAA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFBS	0.044	0.111	0.276	0.553	1.11	2.76	13.8
PFBA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDS	0.048	0.121	0.301	0.603	1.21	3.01	15.1
PFDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFDoDS	0.048	0.121	0.303	0.605	1.21	3.03	15.1
PFDoDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFHpS	0.048	0.119	0.298	0.595	1.19	2.98	14.9
PFHpA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFHxS	0.046	0.114	0.284	0.569	1.14	2.84	14.2
PFHxA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFNS	0.048	0.120	0.300	0.600	1.20	3.00	15.0
PFNA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFOSA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFOS	0.047	0.117	0.291	0.583	1.17	2.91	14.6
PFOA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFPeS	0.047	0.117	0.293	0.586	1.17	2.93	14.7
PFPeA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTeDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFTrDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFUnDA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFMPA	0.100	0.250	0.625	1.250	2.50	6.25	31.3
PFMBA	0.100	0.250	0.625	1.250	2.50	6.25	31.3
NFDHA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFEESA	0.089	0.223	0.556	1.113	2.23	5.56	27.8
NMePFOSAE	0.250	0.625	1.563	3.125	6.25	15.63	78.1
NMePFOSA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
NEtPFOSAE	0.500	1.250	3.125	6.250	12.50	31.25	156.3
NEtPFOSA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
3:3 FTCA	0.250	0.625	1.563	3.125	6.25	15.63	78.1
5:3 FTCA	0.500	1.250	3.125	6.250	12.50	31.25	156.3
7:3 FTCA	0.500	1.250	3.125	6.250	12.50	31.25	156.3
PFECHS	0.046	0.115	0.288	0.576	1.15	2.88	14.4
FBSA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
FHxSA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFPrA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
PFODA	0.050	0.125	0.313	0.625	1.25	3.13	15.6
TFSI	0.049	0.122	0.306	0.612	1.22	3.06	15.3
13C4-PFBA	2.500	2.500	2.500	2.500	2.500	2.500	2.500
13C5-PFPeA	1.250	1.250	1.250	1.250	1.25	1.25	1.25
13C5-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C4-PFHpA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C8-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C9-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C6-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C7-PFUnA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFDoA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFTeDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C3-PFBS	0.581	0.581	0.581	0.581	0.581	0.581	0.581
13C3-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591
13C8-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
13C8-PFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
D3-NMeFOSAA	1.250	1.250	1.250	1.250	1.250	1.250	1.250
D5-NEtFOSAA	1.250	1.250	1.250	1.250	1.250	1.250	1.250
13C2-4:2FTS	1.168	1.168	1.168	1.168	1.168	1.168	1.168
13C2-6:2FTS	1.188	1.188	1.188	1.188	1.188	1.188	1.188
13C2-8:2FTS	1.198	1.198	1.198	1.198	1.198	1.198	1.198
13C3-HFPO-DA	2.500	2.500	2.500	1.250	1.250	1.250	1.250
D7-NMeFOSE	6.250	6.250	6.250	6.250	6.250	6.250	6.250
D9-NEtFOSE	6.250	6.250	6.250	6.250	6.250	6.250	6.250
D5-NEtFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
D3-NMeFOSA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C3-PFBA	1.250	1.250	1.250	1.250	1.250	1.250	1.250
13C4-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C2-PFDA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C4-PFOS	0.598	0.598	0.598	0.598	0.598	0.598	0.598
13C5-PFNA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C2-PFHxA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
18O2-PFHxS	0.591	0.591	0.591	0.591	0.591	0.591	0.591
13C2-PFOA	0.625	0.625	0.625	0.625	0.625	0.625	0.625
13C2-PFUnA	0.313	0.313	0.313	0.313	0.313	0.313	0.313
13C3-PFPeA	1.250	1.250	1.250	1.250	1.250	1.250	1.250

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Attachment 12 - 1633 Bile Salts Stock Solution							
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Aliquot (g)**	**Final Volume of MeOH (mL)**	**Final Conc Bile Salty Stock Soln (ng/mL)**
Sigma Aldrich	T0557-500MG	Taurodeoxycholic acid	516-50-7	TDCA	0.05	50	1000000
Sigma Aldrich	T6260-1G	Taurochenodeoxycholic acid	516-35-8	TCDCA	0.05		950000
Calbiochem	580549-1GM	Tauroursodeoxycholic acid	14605-22-2	TUDCA	0.05		1000000

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Attachment 13 - 1633 Bile Salts Intermediate							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume of MeOH (mL)**	**Final Conc. Bile Salt Intermediate (ng/mL)**
Bile Salts Stock Solution	Taurodeoxycholic acid	516-50-7	TDCA	1000000	1	4	250000
	Taurochenodeoxycholic acid	516-35-8	TCDCA	950000			237500
	Tauroursodeoxycholic acid	14605-22-2	TUDCA	1000000			250000

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Attachment 14 - 1633 Bile Salts Working Solution							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume of MeOH (mL)**	**Final Conc. Bile Salts Working Solution (ng/mL)**
Bile Salts Intermediate	Taurodeoxycholic acid	516-50-7	TDCA	250000	0.1	5	5000
	Taurochenodeoxycholic acid	516-35-8	TCDCA	237500			4750
	Tauroursodeoxycholic acid	14605-22-2	TUDCA	250000			5000

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Attachment 15 - 1633 Window-Defining Matrix Standard							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Stock Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume of MeOH (mL)**	**Final Conc. 1633 WDM Solution (ng/ml)**
Bile Salts Working Solution	Taurodeoxycholic acid	516-50-7	TDCA	5000	0.05	10	25
	Taurochenodeoxycholic acid	516-35-8	TCDCA	4750			23.75
	Tauroursodeoxycholic acid	14605-22-2	TUDCA	5000			25
1633 TOP SS Intermediate	Perfluoro-n-(1,2-13C2)octanoic acid	STL00623	13C2-PFOA	125	0.05		0.625
	Perfluoro-n-(1,2-13C2)undecanoic acid	STL00997	13C2-PFUnA	62.5			0.313
	Perfluoro-n-(3,4,5-13C3)pentanoic acid	STL03269	13C3-PFPeA	250			1.250
1633 EIS Intermediate	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	500	0.0500		2.500
	Perfluoro-n-[13C5]pentanoic acid	STL01893	13C5-PFPeA	250			1.250
	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	13C5-PFHxA	125			0.625
	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	125			0.625
	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	125			0.625
	Perfluoro-n-[13C9]nonanoic acid	STL02578	13C9-PFNA	63			0.313
	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	63			0.313
	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	13C7-PFUnA	63			0.313
	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	63			0.313
	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	63			0.313
	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PFBS	116			0.581
	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	13C3-PFHxS	118			0.591
	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	120			0.598
	erfluoro-1-[13C8]octanesulfonamide	STL01056	13C8-PFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	250			1.250
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	250			1.250
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	234			1.168
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	13C2-6:2FTS	238			1.188
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	13C2-8:2FTS	240			1.198
	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	13C3-HFPO-DA	500			2.500
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	1250			6.250
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	1250			6.250
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	125			0.625
1633 NIS Intermediate	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	250	0.05		1.250
	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	125			0.625
	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	119.5			0.598
	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	62.5			0.313
	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	125			0.625
	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	18O2-PFHxS	118.25			0.591

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Attachment 16 - 1633 Mid-Level Spike							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Stock Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume of MeOH (mL)**	**Final Conc. Native 1633 Mid-Level Spike (ng/ml)**
Native PFAS Intermediate A	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	294	5.0	10.0	147.2
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	291			145.6
	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	118			58.9
	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	94			46.8
	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	479			239.5
	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	292			145.9
	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	474			237.0
	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			62.5
	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			62.5
	Perfluorobutanesulfonic acid	375-73-5	PFBS	111			55.3
	Perfluorobutanoic acid	375-22-4	PFBA	125			62.5
	Perfluorodecanesulfonic acid	335-77-3	PFDS	121			60.3
	Perfluorodecanoic acid	335-76-2	PFDA	125			62.5
	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	121			60.5
	Perfluorododecanoic acid	307-55-1	PFDoDA	125			62.5
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			59.5
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			62.5
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	114			56.9
	Perfluorohexanoic acid	307-24-4	PFHxA	125			62.5
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			60.0
	Perfluorononanoic acid	375-95-1	PFNA	125			62.5
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			62.5
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	117			58.3
	Perfluorooctanoic acid	335-67-1	PFOA	125			62.5
	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	117			58.6
	Perfluoropentanoic acid	2706-90-3	PFPeA	125			62.5
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			62.5
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			62.5
	Perfluoroundecanoic acid	2058-94-8	PFUnDA	125			62.5
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			125.0
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			125.0
	Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	125			62.5
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	223			111.3
	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSAE	625			312.5
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	125			62.5
	2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	1691-99-2	NEtPFOSAE	1250			625.0
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	125			62.5
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			312.5
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	1250			625.0
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	1250			625.0
	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	115			57.6
	Perfluoro-1-butanesulfonamide	30334-69-1	FBSA	125			62.5
	Perfluoro-1-hexanesulfonamide	41997-13-1	FHxSA	125			62.5
	Perfluoropropanoic acid	422-64-0	PFPrA	125			62.5
	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	125			62.5
	Bis(trifluoromethylsulfonyl)amine	82113-65-3	TFSI	122			61.2

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Attachment 17 - 1633 Low-Level Spike							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Stock Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume of MeOH (mL)**	**Final Conc. Native 1633 Low-Level Spike (ng/ml)**
Native PFAS Intermediate A	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	294	0.5	10.0	14.72
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	291			14.56
	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	118			5.89
	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	94			4.68
	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	479			23.95
	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	292			14.59
	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	474			23.70
	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			6.25
	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			6.25
	Perfluorobutanesulfonic acid	375-73-5	PFBS	111			5.53
	Perfluorobutanoic acid	375-22-4	PFBA	125			6.25
	Perfluorodecanesulfonic acid	335-77-3	PFDS	121			6.03
	Perfluorodecanoic acid	335-76-2	PFDA	125			6.25
	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	121			6.05
	Perfluorododecanoic acid	307-55-1	PFDoDA	125			6.25
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			5.95
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			6.25
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	114			5.69
	Perfluorohexanoic acid	307-24-4	PFHxA	125			6.25
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			6.00
	Perfluorononanoic acid	375-95-1	PFNA	125			6.25
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			6.25
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	117			5.83
	Perfluorooctanoic acid	335-67-1	PFOA	125			6.25
	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	117			5.86
	Perfluoropentanoic acid	2706-90-3	PFPeA	125			6.25
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			6.25
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			6.25
	Perfluoroundecanoic acid	2058-94-8	PFUnDA	125			6.25
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			12.50
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			12.50
	Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	125			6.25
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	223			11.13
	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSAE	625			31.25
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	125			6.25
	2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	1691-99-2	NEtPFOSAE	1250			62.50
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	125			6.25
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			31.25
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	1250			62.50
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	1250			62.50
	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	115			5.76
	Perfluoro-1-butanesulfonamide	30334-69-1	FBSA	125			6.25
	Perfluoro-1-hexanesulfonamide	41997-13-1	FHxSA	125			6.25
	Perfluoropropanoic acid	422-64-0	PFPrA	125			6.25
	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	125			6.25
	Bis(trifluoromethylsulfonyl)amine	82113-65-3	TFSI	122			6.12

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Attachment 18 - 1633 ICV Working Standard							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Stock Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume of MeOH (mL)**	**Final Conc. ICV (ng/mL)**
Native PFAS Intermediate A	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	294.375	0.0500	10	1.472
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	291.25			1.456
	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	117.75			0.589
	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	93.5			0.468
	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	479			2.395
	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	291.875			1.459
	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	474			2.370
	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			0.625
	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			0.625
	Perfluorobutanesulfonic acid	375-73-5	PFBS	110.5			0.553
	Perfluorobutanoic acid	375-22-4	PFBA	125			0.625
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120.5			0.603
	Perfluorodecanoic acid	335-76-2	PFDA	125			0.625
	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	121			0.605
	Perfluorododecanoic acid	307-55-1	PFDoDA	125			0.625
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119			0.595
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			0.625
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	113.75			0.569
	Perfluorohexanoic acid	307-24-4	PFHxA	125			0.625
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120			0.600
	Perfluorononanoic acid	375-95-1	PFNA	125			0.625
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			0.625
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	116.5			0.583
	Perfluorooctanoic acid	335-67-1	PFOA	125			0.625
	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	117.25			0.586
	Perfluoropentanoic acid	2706-90-3	PFPeA	125			0.625
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			0.625
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			0.625
	Perfluoroundecanoic acid	2058-94-8	PFUnDA	125			0.625
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			1.250
	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	250			1.250
	Nonafluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	125			0.625
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	222.5			1.113
	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSAE	625			3.125
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	125			0.625
	2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	1691-99-2	NEtPFOSAE	1250			6.250
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	125			0.625
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			3.125
	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	1250			6.250
	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	1250			6.250
	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	115.25			0.576
	Perfluoro-1-butanesulfonamide	30334-69-1	FBSA	125			0.625
	Perfluoro-1-hexanesulfonamide	41997-13-1	FHxSA	125			0.625
	Perfluoropropanoic acid	422-64-0	PFPrA	125			0.625
	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	125			0.625
	Bis(trifluoromethylsulfonyl)amine	82113-65-3	TFSI	122.375			0.612
1633 TOP SS Intermediate	Perfluoro-n-(1,2-13C2)octanoic acid	STL00623	13C2-PFOA	125	0.05		0.625
	Perfluoro-n-(1,2-13C2)undecanoic acid	STL00997	13C2-PFUnA	62.5			0.313
	Perfluoro-n-(3,4,5-13C3)pentanoic acid	STL03269	13C3-PFPeA	250			1.250
1633 EIS Intermediate	Perfluoro-n-[13C4]butanoic acid	STL00992	13C4-PFBA	500	0.05		2.500
	Perfluoro-n-[13C5]pentanoic acid	STL01893	13C5-PFPeA	250			1.250
	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	13C5-PFHxA	125			0.625
	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	13C4-PFHpA	125			0.625
	Perfluoro-n-[13C8]octanoic acid	STL01052	13C8-PFOA	125			0.625
	Perfluoro-n-[13C9]nonanoic acid	STL02578	13C9-PFNA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	13C6-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	13C7-PFUnA	62.5			0.313
	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	13C2-PFDoA	62.5			0.313
	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	13C2-PFTeDA	62.5			0.313
	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	13C3-PFBS	116.25			0.581
	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	13C3-PFHxS	118.25			0.591
	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	13C8-PFOS	119.5			0.598
	erfluoro-1-[13C8]octanesulfonamide	STL01056	13C8-PFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D3-NMeFOSAA	250			1.250
	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D5-NEtFOSAA	250			1.250
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	13C2-4:2FTS	233.5			1.168
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	13C2-6:2FTS	237.5			1.188
	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	13C2-8:2FTS	239.5			1.198
	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	13C3-HFPO-DA	500			2.500
	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D7-NMeFOSE	1250			6.250
	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D9-NEtFOSE	1250			6.250
	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D5-NEtFOSA	125			0.625
	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D3-NMeFOSA	125			0.625
1633 NIS Intermediate	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	13C3-PFBA	250	0.05		1.250
	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	13C4-PFOA	125			0.625
	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	13C2-PFDA	62.5			0.313
	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	13C4-PFOS	119.5			0.598
	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	13C5-PFNA	62.5			0.313
	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	13C2-PFHxA	125			0.625
	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	18O2-PFHxS	118.25			0.591

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Attachment 19

Purchased Standards					
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/ml)
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnA	50000
Wellington	MPFDOA	Perfluorododecanoic acid	307-55-1	PFDoA	50000
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200
Wellington	L-PFPeS	Perfluoropentansulfonic acid	2706-91-4	PFPeS	46900
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46600
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoS	48400
Wellington	4;2 FTS	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2-FTS	46700
Wellington	6;2 FTS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2-FTS	47400
Wellington	8;2 FTS	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2-FTS	47900
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000
Wellington	br-NMeFOSA	N-methyl perfluorooctanesulfonamide	31506-32-8	NMePFOSA	50000
Wellington	br-NEtFOSA	N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtPFOSA	50000
Wellington	br-NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000
Wellington	br-NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000
Wellington	br-NMeFOSE	N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMePFOSAE	50000
Wellington	br-NMeFOSE	N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtPFOSAE	50000
Wellington	HFPO-DA	Hexafluoropropylene oxide dimer acid	13252-13-6	HFPODA	50000
Wellington	NaDONA	4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	DONA	47100
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500
Wellington	PF4OPeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000
Wellington	PF5OHxA	Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	50000
Wellington	3,6-OPFHpA	Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	50000
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000
Wellington	FPePA	2H,2H,3H,3H-Perfluorooctanoic acid	914637-49-3	5:3 FTCA	50000
Wellington	FHpPA	3-Perfluoroheptyl propanoic acid	812-70-4	7:3 FTCA	50000
Wellington	PFECHS	Perfluoro-4-ethylcyclohexanesulfonate	335-24-0	PFECHS	46100
Wellington	FBSA-I	Perfluoro-1-butanesulfonamide	30334-69-1	FBSA	50000
Wellington	FHxSA-I	Perlfuoro-1-hexanesulfonamide	41997-13-1	FHxSA	50000
Wellington	PFPrA	Perfluoropropanoic acid	422-64-0	PFPrA	50000
Wellington	PFODA	Perfluoro-n-octadecanoic acid	16517-11-6	PFODA	50000
AccuStandard	PFOS-030S	Bis(trifluoromethylsulfonyl)amine	82113-65-3	TFSI	97900
Wellington	MPFBA	Perfluoro-n-[13C4]butanoic acid	STL00992	$^{13}C_4$-PFBA	50000
Wellington	M5PFPeA	Perfluoro-n-[13C5]pentanoic acid	STL01893	$^{13}C_5$-PFPeA	50000
Wellington	M5PFHxA	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	STL02577	$^{13}C_5$-PFHxA	50000
Wellington	M4PFHpA	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	STL01892	$^{13}C_4$-PFHpA	50000
Wellington	M8PFOA	Perfluoro-n-[13C8]octanoic acid	STL01052	$^{13}C_8$-PFOA	50000
Wellington	M9PFNA	Perfluoro-n-[13C9]nonanoic acid	STL02578	$^{13}C_9$-PFNA	50000
Wellington	M6PFDA	Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	STL02579	$^{13}C_6$-PFDA	50000
Wellington	M7PFUdA	Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	STL02580	$^{13}C_7$-PFUnA	50000
Wellington	MPFDoA	Perfluoro-n-[1,2-13C2]dodecanoic acid	STL02703	$^{13}C_2$-PFDoA	50000
Wellington	M2PFTeDA	Perfluoro-n-[1,2-13C2]tetradecanoic acid	STL02116	$^{13}C_2$-PFTeDA	50000
Wellington	M3PFBS	Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	STL02337	$^{13}C_3$-PFBS	46500
Wellington	M3PFHxS	Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	STL02581	$^{13}C_3$-PFHxS	47300
Wellington	M8PFOS	Perfluoro-1-[13C8]octanesulfonic acid	STL01054	$^{13}C_8$-PFOS	47800
Wellington	M8FOSA-I	Perfluoro-1-[13C8]octanesulfonamide	STL01056	$^{13}C_8$-PFOSA	50000
Wellington	d3-N-MeFOSAA	N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	D_3-NMeFOSAA	50000
Wellington	d5-N-EtFOSAA	N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	D_5-NEtFOSAA	50000
Wellington	M2-4;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid	STL02395	$^{13}C_2$-4:2FTS	46700
Wellington	M2-6;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid	STL02279	$^{13}C_2$-6:2FTS	47500
Wellington	M2-8;2FTS	1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid	STL02280	$^{13}C_2$-8:2FTS	47900
Wellington	M3HFPO-DA	Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	STL02255	$^{13}C_3$-HFPO-DA	50000
Wellington	d7-N-MeFOSE-M	N-methyl-d7-perfluorooctanesulfonamidoethanol	STL02277	D_7-NMeFOSE	50000
Wellington	d9-N-EtFOSE-M	N-ethyl-d9-perfluorooctanesulfonamidoethanol	STL02278	D_9-NEtFOSE	50000
Wellington	d-N-MeFOSA-M	N-ethyl-d5-perfluoro-1-octanesulfonamide	STL02704	D_5-NEtFOSA	50000
Wellington	d-N-EtFOSA-M	N-methyl-d3-perfluoro-1-octanesulfonamide	STL02705	D_3-NMeFOSA	50000
Wellington	M3PFBA	Perfluoro-n-[2,3,4-13C3]butanoic acid	STL02680	$^{13}C_3$-PFBA	50000
Wellington	MPFOA	Perfluoro-n-[1,2,3,4-13C4]octanoic acid	STL00990	$^{13}C_4$-PFOA	50000
Wellington	MPFDA	Perfluoro-n-[1,2-13C2]decanoic acid	STL00996	$^{13}C_2$-PFDA	50000
Wellington	MPFOS	Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	STL00991	$^{13}C_4$-PFOS	47800
Wellington	MPFNA	Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid	STL00995	$^{13}C_5$-PFNA	50000
Wellington	MPFHxA	Perfluoro-n-[1,2-13C2]hexanoic acid	STL00993	$^{13}C_2$-PFHxA	50000
Wellington	MPFHxS	Perfluoro-1-hexane[18O2]sulfonic acid	STL00994	$^{18}O_2$-PFHxS	47300
Wellington	M2PFOA	Perfluoro-n-(1,2-13C2)octanoic acid	STL00623	$^{13}C_2$-PFOA	50000
Wellington	MPFUdA	Perfluoro-n-(1,2-13C2)undecanoic acid	STL00997	$^{13}C_2$-PFUnA	50000
Wellington	M3PFPeA	Perfluoro-n-(3,4,5-13C3)pentanoic acid	STL03269	$^{13}C_3$-PFPeA	50000
Sigma Aldrich	T0557-500MG	Taurodeoxycholic acid	516-50-7	TDCA	neat
Sigma Aldrich	T6260-1G	Taurochenodeoxycholic acid	516-35-8	TCDCA	neat
Calbiochem	580549-1GM	Tauroursodeoxycholic acid	14605-22-2	TUDCA	neat

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eurofins Document number: **T-PFAS-WI49209** Old Reference:	Always check on-line for validity. **Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633**	Level: **Work Instruction**
Version: **4**		Organisation level: **5-Sub-BU**
Approved by: **ULJC** Effective Date: **21-DEC-2023**	Document users: **5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep**	Responsible: **5_EUUSLA_PFAS_Manager**

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Revision Log

	Revision: 04	**Effective date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout document	Requirement	Added references to Draft 4. Removed references to Sciex 4500.
Personnel Training and Qualification	Enhancement	Corrected table and version references
Sample Collection, Preservation, and Handling	Enhancement	added statement regarding landfill leachates
Apparatus and Equipment	Clarification	Updated to correct Cat#s, Corrected WAX cartridge

	Revision: 04	**Effective date:** This version
Section	**Justification**	**Changes**
Reagents and Standards	Clarification	Updated to correct Cat#s
Reagents and Standards	Method Requirement	Corrected % purity
Calibration A	Method Requirement	Added S/N for compounds without confirmation ions Removed verbiage regarding Linear/Branched standard
Calibration A	Clarification	Removed WV requirements
Procedure B/D	Clarification	Corrected the Envi-carb and filtration steps done in in the instrument room. Corrected IS name.
Procedure E	Method Requirement	Instrument Blanks report to MDL
Procedure E	Enhancement	Added reporting procedure for samples with failing EIS and NIS. Updated RT criteria.
Procedure E	Clarification	Updated NIS criteria
Procedure E	Clarification	Mass tune must be +/- 0.2 amu
Statistical Information/Method Performance	Clarification	Updated NIS criteria
Attachment 3	Enhancement	Updated to reflect current method
Attachment 5-20	Enhancement	updated attachments to reflect new standards

Revision:	3	**Effective date:** 09.MAY.2023
Section	**Justification**	**Changes**
Revision Log	Formatting Requirement	Revision logs up to the previous version
Reference	Enhancement	Update to version 3 of the Draft method
Personnel Training and Qualifications	Enhancement	Add requirements from Table 5 for IPR(IDOC)
Apparatus and Equipment	Enhancement	Proprietary
Calibration	Reflect current procedure	replace TDCA with Bile Salts as appropriate
Procedure	Enhancement	Add option for automated SPE extraction
Procedure	Reflect current procedure	replace TDCA with bile Salts as appropriate;clarified process when IDAs fail. Add verbiage about multiple cartridge prep and samples that require centrifugation. Add sample duplicate requirement
Reagents and Standards/attachment 13-18	Reflect current procedure	reflect current preparation and standard names.
Statistical Information/Method Performance	Reflect current requirement	QC windows are method defined add reference tables from 1633 draft V3. add sample duplicate requirement

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Reference

1. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, 4th DRAFT, July 2023.

2. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, 3rd DRAFT, December 2022.

3. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, 2nd DRAFT, June 2022.

4. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, DRAFT, August 2021.

5. Chemical Hygiene Plan, current version.

Cross Reference

Document	Document Title
T-PFAS-WI21568	Manifold and N-EVAP Cleaning for PFAS Extractions
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses
T-WC-WI53304	Total Suspended Solids (TSS Gravimetric) Prescreen by EPA Draft Methods in Aqueous Samples
QA-SOP11178	Demonstrations of Capability
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
G-DC-FRM23907	Redacted SOPs

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in aqueous samples to include non-potable waters and non-regulatory potable water when directed by the client. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS. Compounds other than those listed may be analyzed by client request.

Analyte	Acronym	CAS#
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1

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Analyte	Acronym	CAS#
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnDA	2058-94-8
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
8:2 - Fluorotelomersulfonic acid	8:2FTS	39108-34-4
N-methylperfluoro-1-octanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethylperfluoro-1-octanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
4:2-Fluorotelomersulfonic acid	4:2-FTS	757124-72-4
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
6:2-Fluorotelomersulfonic acid	6:2-FTS	27619-97-2
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoDS	79780-39-5
Perfluorooctanesulfonamide	PFOSA	754-91-6
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMePFOSAE	24448-09-7
N-methylperfluoro-1-octanesulfonamide	NMePFOSA	31506-32-8
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtPFOSAE	1691-99-2
N-ethylperfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid; (Hexafluoropropylene oxide dimer acid)	HFPODA	13252-13-6
Ammonium 4,8-dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS, F53B major	756426-58-1 *
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS, F53B minor	763051-92-9 *
3-Perfluoropropylpropanoic acid	3:3 FTCA	356-02-5
3-Perfluoropentylpropanoic acid	5:3 FTCA	914637-49-3
3-Perfluoroheptylpropanoic acid	7:3 FTCA	812-70-4
Perfluoro-3-methoxypropanoic acid	PFMPA, PFECA F	377-73-1

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Analyte	Acronym	CAS#
Perfluoro-4-methoxybutanoic acid	PFMBA, PFECA A	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA, PFECA B	151772-58-6
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA, PES	113507-82-7

*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

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Basic Principles

An aqueous sample is fortified with isotopically-labeled extraction standards and is passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. Carbon cleanup is performed on each sample extract. The extract is filtered and fortified with Isotopically-labeled injection internal standards. It is then analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where possible.

2. Proprietary Content

3. All part of the SPE manifold must be cleaned as per *T-PFAS-WI21568*.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as “likely to be carcinogenic to humans”. Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. IDOC trials are spiked at the OPR Level. IDOC must meet the requirements for the IPR as listed in Tables 5 and 6 of method 1633 draft version 4. See *Attachment 21*.

See *QA-SOP11178* for additional information on IDOC and DOC.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 500-mL HDPE containers. For sources that are known or expected to contain high levels of PFAS (e.g. landfill leachates), samples may be collected in 125-mL HDPE containers, provided regulatory limits are met. All sample containers must have linerless HDPE or polypropylene caps. Keep the sample sealed from time of collection until extraction. The entire aqueous sample that is collected must be prepared and analyzed, and subsampling must be avoided whenever possible.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

B. Sample Storage and Shipment

1. Samples must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when the samples are

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received at the laboratory.

2. Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, and protected from light until extraction. Alternatively, to meet project requirements, samples may be stored at less than or equal to -20°C and protected from light until extraction.

3. Water samples must be extracted within 28 days when stored at a temperature of 0° to 6°C, not frozen, and protected from light. Water samples must be extracted within 90 days when stored at a temperature less than or equal to -20°C and protected from light. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C and protected from light.

Apparatus and Equipment

A. Apparatus

1. 500-mL HDPE bottles: CapSure Leak-Proof bottles, Catalog No. 0500-1902-QC or equivalent

2. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Catalog No. 05-539-5 or equivalent

3. 10-mL polypropylene volumetric flask, Class A – Fisher Scientific, Catalog No. S02288 or equivalent.

4. HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Catalog No. 1000-1902-PC.

5. Analytical Balance – Capable of weighing to 0.0001 g

6. Top-Loading Balance – Capable of weighing to 0.01 g

7. Solid phase extraction (SPE) Weak Anion Exchange ("WAX") cartridge – Phenomenex; Strata-X-AW 33 µm Polymeric Weak Anion, 200 mg / 6 mL, Tubes; Catalog No. 8B-S038-FCH or equivalent.

8. Large-volume SPE Reservoir (25-mL) - Millipore-Sigma, Product No. 54258-U.

9. SPE Tube Adapter - Millipore-Sigma, Catalog No. 57020-U.

10. SPE vacuum extraction manifold –"Resprep" 24-port manifold. Restek, Catalog No. 26080 or equivalent.

11. Polypropylene SPE delivery needles – Agilent, Catalog No. 12234511.

12. Centrifuge – "Q-Sep 3000"; Restek Corp. Catalog No. 26230 or equivalent, capable of a minimum rotational speed of 3000 rpm.

13. Auto Pipettes – Eppendorf, Repeater; capable of accurately dispensing 10- to 1000-µL. Fisher Scientific Catalog No. 14-287-150 or equivalent.

14. Polypropylene pipette tips - 0.1-200 µL. Fisher Scientific, Catalog No. 02-681-135

15. Polypropylene pipette tips - 100-1000µL. Fisher Scientific, Catalog No. 02-707-507

16. Pipettes – Disposable transfer. Fisher Scientific, Catalog No. 13-711-7M

17. Vortex mixer - variable speed. Fisher Scientific or equivalent.

18. N-Evap sample extract concentrator with N_2supply and water bath for temperature control. Organomation, Inc. Catalog No. 11250 or equivalent.

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19. Reagent Water Purification System: Capable of producing ultrapure "Type 1/Milli-Q"-grade water from in-house deionized water system. Millipore SAS; Catalog No. FTPF08831.

20. SureStart Polyspring inserts, catalog number 03452340

21. Short-Cap Vial with Grad Marking Spot, 9-425 Screw-Thread, 2.0 mL, 9 mm, Clear, 1000-pk., Restek Catalog No. 21141 or equivalent.

22. Short Screw Caps, Polypropylene, Screw-Thread, PTFE/Silicone Septa, Blue, Preassembled, 2.0 mL, 9 mm, 100-pk, Restek Catalog No. 24485 or equivalent.

23. Centrifuge tubes – 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Catalog No. 06-443-21 or equivalent

24. Polypropylene bottles for standard storage – 4 mL; Fisher Scientific, Catalog No. 02-923-11E or equivalent.

25. Stainless steel spatula/scoop set. Bel-Art SP Scienceware; Product No. 11-865-130 or equivalent.

26. pH paper - Hydrion, range 5.5-8.0. Fisher Scientific, Catalog No. 14-853-275 or equivalent

27. Syringe filter - Acrodisc, Syringe Filter, GHP,13 mm, 0.2 µm, Aqueous, 100/pkg, Part No. WAT097962.

28. Silanized glass wool (Sigma-Alrich, Catalog No. 20411 or equivalent

29. Disposable syringe filter, 25-mm, 0.2um Nylon membrane, PALL/Acrodisc or equivalent

30. Glass fiber filter, 47 mm, 1 um, PALL A/E or equivalent

31. Wheaton Bottle, 15ml, narrow mouth, HDPE, Leak resistant; DWK Life Sciences, Catalog No. 209044SP or equivalent

32. Graduated Medicine Cups, Plastic, 30mL. Medline Industries Catalog No. DYND8000 or equivalent.

B. Equipment

1. AB Sciex Triple Quad 5500/5500 Plus Turbo V Ion Source or equivalent

   ExionLC Controller
   ExionLC AC Pump
   ExionLC AC Autosampler
   Exion AC Column Oven
   Data system – Analyst 1.6.3

2. Proprietary Content

3. HPLC columns

   a. Proprietary Content

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All

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non-solvents are stored according to manufacturer's storage conditions.

A. Reagents

1. Methanol (MeOH) – VWR "Chromasolv LC-MS" grade Cat. No. BJ34966-4L or equivalent
2. Acetonitrile (ACN) – Fisher Scientific, Optima Cat. No. A955-4 or equivalent
3. Ammonium acetate – Sigma Aldrich, Cat. No. 431311-250G or equivalent. Store at 2-8°C.
4. Ammonium hydroxide, 30% in water, certified ACS+ grade or equivalent, store at room temperature
5. Aqueous ammonium hydroxide (3%) – add ammonium hydroxide (10 mL, 30%) to reagent water (90 mL). Store at room temperature. Replace after 3 months.
6. Methanolic ammonium hydroxide (1%) - add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month
7. Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid - add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month.
8. Acetic Acid – ACS grade. Store at room temperature. Sigma Aldrich Cat. No. 695092-100mL or equivalent.
9. Acetic Acid (0.1%) – dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.
10. Formic Acid
    a. Formic acid (aqueous, 0.1 M) - dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years
    b. Formic acid (aqueous, 0.3 M) - dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years
    c. Formic acid (aqueous, 5% v/v) - mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years
    d. Formic acid (aqueous, 50% v/v) - mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years
    e. Formic acid (methanolic 1:1, 0.1 M formic acid/methanol) - mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years
11. "Superclean Envi-Carb"; bulk sorbent. Supelco Cat# 57210-U or equivalent.
12. 20 mM ammonium acetate solution in 95:5(v/v) Milli-Q water/acetonitrile-Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 1900 mL Milli-Q water and mix well to dissolve the ammonium acetate. Add 100 mL acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.
13. 20 mM ammonium acetate solution in 90:10 acetonitrile/Milli-Q water – Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 200 mL of Milli-Q water and mix well to dissolve the Ammonium Acetate. Add 1800 mL of acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.

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B. Standards

Standards are prepared using calibrated pipettes, polypropylene microcentrifuge tubes, polypropylene bottles, and 10-mL Class A PP volumetric flasks to create solutions at desired concentrations. The concentrated solution is injected below the surface of the diluting solvent After preparation is completed, standards should be vortexed to ensure complete mixing. Measurement of volumes less than 5 µl should be avoided in routine production operations.

All stock, intermediate and spiking solutions are prepared using Methanol.

All initial calibration, initial calibration verification, and linear branched working standard solutions are prepared using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid.

All diluted solutions must be stored in HDPE containers that have been thoroughly rinsed with methanol.

Stock standard and intermediate standard solutions are stored in the refrigerator in labeled polypropylene screw-top vials, PP bottles, or PP centrifuge tubes.

Expiration dates are managed through LIMS Reagent. Solutions transferred from sealed glass ampules to screw-capped vials are given expiration dates of 1 year from the date opened or the expiration date provided by the vendor, whichever occurs sooner. Intermediate solutions are given an expiration date of 6 months from the preparation date, or the expiration date from the ampule provided by the vendor, whichever occurs sooner. The ampules and transferred solutions are stored in the refrigerator.

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator. When these solutions are prepared they must be tested prior to use in the PFAS extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

Working initial calibration solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working initial calibration solution, whichever occurs sooner.

The primary/preferred standard vendor is Wellington Laboratories, Inc. Ontario, Canada. Listed catalog numbers are taken from Wellington product lists. Equivalent standards may be substituted, if the listed standards are unavailable.

The solution concentration listed is as presented on the certificate of analysis and includes adjustment for purity and the salt form of the compound used.

The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See *Attachment 4*. If the compound purity is assayed to be 98% or greater, weight can be used without correction to calculate concentrations.

Log purchased standards into LIMS Reagent. Select the solution category SOURCE for purchased mixes and/or single-compound ampules. LIMS Reagent system will assign formatted names to the purchased standard solutions. The automatically-generated name can be overwritten with a manually created name if desired. Use labels printed through the LIMS Reagent to identify and track standard solutions after transfer from original ampule to storage vial. The CofA for the ampulated stock standard is attached in LIMS Reagent for reference.

Standards are prepared by transferring a known quantity of Standard to a final volume of solvent. Standard Preparation is documented in LIMS Reagent. Solutions are stored by Type in LIMS Reagent,

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i.e., INTERMEDIATE=working solutions and intermediate standards and SOURCE=stocks (ampulated solutions). Each Standard is given a unique name.

The following attachments provide examples of standard preparation and purchasing information. Refer to the documentation in LIMS Reagent for standards preparation information.
Attachment 5 - 1633 Native PFAS Intermediate A
Attachment 6 - 1633 Native PFAS Intermediate B
Attachment 7 - 1633 Extracted Internal Standard Intermediate
Attachment 8 - 1633 Non-Extracted Internal Standard Intermediate
Attachment 9 - 1633 TOP Surrogate Intermediate
Attachment 10 - 1633 ICAL Standard Preparation Scheme
Attachment 11 - 1633 Initial Calibration Standard Concentrations
Attachment 12 - Bile Salts Stock Solution
Attachment 13 - Bile Salts Intermediate
Attachment 14 - Bile Salts Working Solution
Attachment 15 - 1633 WDM Standard
Attachment 16 - 1633 Native Mid-Level Spike
Attachment 17 - 1633 Native Low-Level Spike Intermediate
Attachment 18 - 1633 Native Low-Level Spike
Attachment 19 - 1633 ICV Working Standard
Attachment 20 - 1633 Purchased/Ampulated Standards

Calibration

A. Initial Calibration

1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order curve fit (quadratic). In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, NEtFOSAA, PFNA, PFOA, PFOSA, NEtFOSE, NMeFOSE, NEtFOSA, and NMeFOSA. S/N ratio for the lowest standard within the quantitation range must be greater than or equal to 3:1 for quantification ions and the confirmation ions, or greater than or equal to 10:1 if the analyte only has a quantification ion.

2. Analyze a Cal4 level standard that contains Bile Salt retention time markers. The analysis of this standard is used to evaluate the interference from bile salts in samples.

   Example Initial Calibration Sequence:
   1. Instrument Blank
   2. Instrument Blank
   3. Instrument Blank
   4. CAL 1
   5. CAL 2
   6. CAL 3
   7. CAL 4
   8. CAL 5
   9. CAL 6
   10. CAL 7
   11. ICB (Instrument Blank)
   12. ICV
   13. WDM (Bile Salts standard)

3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See *Attachment 2* for additional information about compound relationships.

4. Analyze a standard containing 100 ppb of Taurodeoxycholic Acid (TDCA), Taurochenodeoxycholic Acid (TCDCA), and Tauroursodeoxycholic Acid (TUDCA). The analysis of this standard is used to

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evaluate the chromatographic program relative to the risk of an interference from bile salts in samples. The analytical conditions must be set to allow a separation of at least 1 minute between TDCA and PFOS.

Note: For better accuracy, PFTrDA is quantitated using the average of the areas of labeled compounds 13C2-PFTeDA and 13C2-PFDoDA.

Compound	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS

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Compound	Extraction Standard
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

5. Fit the curve.

   a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.

   b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%.

   c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within ±30% of its true value.

   d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

   NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See *Attachment 4*.

6. Initial Calibration Verification (ICV)

   A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock by a second analyst may be used. The calculated amount for each analyte must be within ±30% of the true value. If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.

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2. Acceptance criteria

    a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

    b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

All water samples should be evaluated for Total Suspended Solids (TSS) as per *T-WC-WI53304*. Although the full container volume of sample should be extracted, this method is applicable to aqueous samples containing up to 100mg/L of suspended solids. For samples containing > 100mg/l of suspended solids, or when unavoidable due to high levels of PFAS, smaller samples volumes may be analyzed. However, ultimately, the TSS results, visual inspection, and analyst experience are all used to determine the volume to be used to extract the sample.

A. Sample Preparation

1. Weigh sample container with contents on a calibrated top loading balance, and record the first reading in the automated prep entry system.

    a. For all samples, the full bottle must be extracted. The sample must remain in the original sample container until after spiking solutions have been added.

    b. If limited sample is submitted, spike sample in original container, then add Milli-Q water to bring to final volume of 500 mL prior to SPE extraction (see B.6 for spiking details).

    c. If the sample has dissolved and/or settleable solids content (i.e; is cloudy or has a layer of sediment/solids at the bottom of the bottle), the sample must be centrifuged in order to minimize the difficulty of passing through the SPE sorbent bed. In order to preserve the integrity of the sample and ensure the full volume of the container is used, see Procedure C.

2. Use a 500 mL HDPE bottle for the method blank, the laboratory control sample (LCS) and the low level laboratory control sample (LLCS). Fill each bottle with 500 mL of Milli-Q water. Record 500 mL as the volume for the batch QC samples on the batchlog.

3. Check that the pH is 6.5 ± 0.5. If necessary, adjust the pH with 50% formic acid or ammonium hydroxide (or with 5% formic acid and 3% aqueous ammonium hydroxide). **NOTE: Never put pH test strips into the sample. Use a disposable pipette to place a few drops of sample onto the pH paper.**

B. Solid Phase Extraction (SPE) - Manual Sample Extraction

1. Pack clean silanized glass wool to half the height of the WAX SPE cartridge barrel.

2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.

3. Condition each SPE cartridge with the following reagents in the following order without allowing the cartridges to go dry:

    a. 15 mL 1% methanolic ammonium hydroxide

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b. 5 mL 0.3M formic acid

c. Discard conditioning eluent(s)

4. Label each sample bottle, cap and reservoir with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).

5. Vortex all spike solutions prior to use.

6. Spike QC and all samples with 25 µl of Working labeled extraction standard spike solution (PFC_1633_SS_XXXXX). Spike LCS/MS/MSD with 200 µl of native 1633 mid-level spike solution (PFC_1633_MID_XXXXX). Spike LLCS with 400 µl of native 1633 low-level spike solution (PFC_1633_LOW_XXXXX). Vortex/Shake containers to mix thoroughly.

7. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges.

8. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by 5 mL 1:1 0.1M formic acid/methanol and pass the rinses through the cartridge using vacuum. Apply full vacuum (not exceeding 20" Hg) to the cartridges and dry until they are visually similar to an unused cartridge. This may take up to 15 minutes for some cartridges. Discard the rinse solution.

NOTE: If a given sample is too problematic to extract using one cartridge with full vacuum, a second cartridge is created following Procedure B.1-3 above.

9. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge ensuring the delivery needles to do not touch the sides of the tubes.

10. Rinse the inside of each empty sample/QC bottle with 5mL of 1% methanolic ammonium hydroxide. Add to reservoir and cartridge.

11. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.

12. Disconnect the cartridge/adapter from the manifold. Remove the collection tubes.

Note: If a second cartridge was required, the elutant from the second cartridge is added to the first cartridge elutant with a methanol rinse and then blown down to 5 mL on an N-evap prior to step 13 below.

13. Add 25 µL of concentrated acetic acid to each collection tube and vortex to mix.

14. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system. The prep entry system will calculate the sample weight. Record the calculated weight as the sample volume on the batchlog.

Note: The instrument lab chemist performs the next steps.

15. Bring each sample extract to final volume 5 mL using methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

16. Add 10 mg of Superclean Envi-Carb to each sample and batch QC extracts using a 10 mg scoop.

17. Handshake occasionally for no more than 5 minutes. Immediately vortex and centrifuge for 10 minutes.

18. Add 25 µL of Internal Standard Spike Solution (PFC_1633_IS_XXXXX) to a clean 15-mL polypropylene centrifuge collection tube.

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19. Using a 5-mL polypropylene syringe, pull up 5 mL of methanol. Place a syringe filter (25-mm filter, 0.2-um nylon membrane) on the syringe and dispense the filter the methanol to waste. Remove the filter, pull up the plunger of the syringe, place the rinsed filter back on the syringe, and push air through the filter. Repeat this step at least 2 times.

20. Once the filter is dry, pour the supernatant of the sample extract into a medicine cup. Pull up the full volume of sample extract from the medicine cup using the syringe. Place the dried filter back on the syringe and filter the entire extract into the new collection tube containing the internal standard.

21. Cap and vortex to mix.

22. Transfer a portion of the final extract to the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

23. Cap the centrifuge tube. The remaining centrifuged extracts are stored in the refrigerator for dilution or reinjection if needed.

C. Samples containing Dissolved and/or Settleable Solids

1. Check that the pH is 6.5 ± 0.5. If necessary, adjust the pH with 50% formic acid or ammonium hydroxide (or with 5% formic acid and 3% aqueous ammonium hydroxide). **NOTE: Never put pH test strips into the sample. Use a disposable pipette to place a few drops of sample onto the pH paper.**

2. Spike sample with appropriate spikes as in Procedure B.6.

3. Centrifuge the full bottle.

4. DO NOT SHAKE BOTTLE FOLLOWING THE CENTRIFUGE STEP.

5. Follow steps in Procedure B.1 through 4.

6. Attach a 25-mL SPE reservoir to each cartridge. Decant the centrifuged sample onto its respective SPE cartridge. Allow the full volume to pass through each cartridge by gravity if possible. Apply a light vacuum if necessary. The drip rate should be approximately 1-2 drops per second.

7. Rinse the sample bottle with 5mL of Milli-Q water, add the rinseate to the cartridge, and repeat.

8. Continue extraction process with Procedure B.8.

D. Solid Phase Extraction (SPE) – Proprietary Content

1. Proprietary Content

2. Proprietary Content

3. Proprietary Content

4. Proprietary Content

5. Proprietary Content

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6. Proprietary Content
7. Proprietary Content
8. Proprietary Content
9. Proprietary Content
10. Proprietary Content
11. Proprietary Content
12. Proprietary Content
13. Proprietary Content
14. Proprietary Content
15. Proprietary Content
16. Proprietary Content
17. Proprietary Content
18. Proprietary Content
19. Proprietary Content
20. Proprietary Content

E. LC/MS/MS Analysis

1. Mass Calibration and Tuning

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a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

b. When masses fall outside of the ±0.2 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.2 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.

2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (5500/5500 Plus/7500) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

3. Example acquisition method: See *Attachment 3*. Mass Transitions: See *Attachment 1*.

4. Instrument Sensitivity Check (ISC) and Instrument Blanks

   a. Prior to sample analysis, an Instrument Sensitivity Check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within ±30% of their true values. The signal-to-noise ratio must be greater than or equal to 3:1 for the quantitation ions and confirmation ions, and 10:1 for quantitation ions that have no confirmation ion. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.

   b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily at the start of a sequence. The concentration of all analytes must be less than the MDL. If acceptance criteria are not met, the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.

5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.

6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, WDM standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes Bile Salt RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

Example Sample Sequence:

1. Instrument blank
2. Instrument blank
3. Instrument blank
4. Instrument Sensitivity Check (CCV0 _CAL1)
5. CCV 2_CAL4
6. WDM (Bile Salts marker)
7. Instrument Blank (ICB)
8. Method Blank (MB)
9. Low Level LCS (LLCS)
10. LCS
11. Sample (10 or less)
12. CCV 3_CAL4
13. Instrument Blank

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7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extracted method blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted. If there is no target analyte greater than the reporting limit in samples associated with an unacceptable method blank, the data may be reported with qualifiers.

9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.

    a. LLCS, LCS, MS, extraction standard recoveries, and RPDs are calculated and compared to the limits stored on the LIMS.

    b. If LLCS and LCSD recoveries are acceptable, proceed to sample quantitation.

    c. If the LCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, consult supervisor for course of action based on batch and samples results.

    d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

10. Calculate the relative percent difference (RPD) between the sample and the matrix duplicate (DUP). The RPD must be less than or equal to 30%. This criteria only applies to analytes whose concentration in the sample is greater than or equal to the LOQ. If the criteria is not met, narrate with an NCM and report the data.

11. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, MS/MSD, and sample duplicates prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria defined in Table 6 of EPA 1633 Draft 4. Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-noise ratio is greater than 10:1.

12. If the extraction standard recovery(ies) is(are) outside the QC limit(s), dilute the sample extract and re-analyze. If the EIS recovery(ies) of the diluted sample extract are below 5%, a reduced sample aliquot will be extracted. If the EIS recovery(ies) of the diluted sample extract are greater than 5%, the data is reported.

13. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 50-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 50-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.

14. Compare the retention times of all of the analytes, surrogates, and internal standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than 0.4 minutes. Furthermore, native compounds with an exact isotopically-labeled analogue must elute within 0.1 minutes from their associated analogue.

15. If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.

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16. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of PFBA, PFECA F(PFMPA), PFECA A(PFMBA), PFPeA, NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required.

17. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of internal standard spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%.Extracts requiring dilutions greater than 10X should be re-extracted using a reduced aliquot.

Dilution Example 1/10: Mix 895 µL of Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution with 100 µl of sample extract and 5 µl of internal standard spike solution. Vortex to mix. Using an auto-pipette, transfer an aliquot of the mixed solution into a labeled auto-sampler vial. Cap and vortex thoroughly to mix.

Calculations

1. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

   On-column Concentration = peak area ratio ÷ AVE RRF

3. On-Column Analyte Concentration using linear curve

   On-column Concentration = (peak area ratio - intercept) ÷ slope

4. Sample Concentration

   Sample concentration (ng/l) = (On-column concentration x Final Sample Volume x DF) ÷ Initial Sample Volume

5. Ion Ratio

   ion ratio = (peak area or height of quantifier)/(peak area or height of qualifier)

6. See *T-PEST-WI9847* for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

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The LCS (OPR) and LLCS (LLOPR) should contain all compounds of interest. LCS, LLCS, MS, and extraction standard recoveries are compared to the limits stored on the LIMS. These limits are method defined. The LCS (OPR) and LLCS (LLOPR) and their respective EIS limits are from Table 5 and Table 6 of Draft 1633, V4. See *Attachment 21*. The MS/MSD native recoveries are compared to the OPR limits listed in Table 5 Draft 1633, V4

QC parameter	Lower acceptance limit	High acceptance limit
Non-extracted Internal Standard (NIS)	50% of the average NIS from the initial calibration	200%
Sample/Duplicate	NA	less than or equal to 30%

Historical data for MS/Ds, LCSs, EIS is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to *QA-SOP11892* for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank, an LCS/LLCS (Milli Q water spiked with all compounds to be determined carried through the entire procedure), and a sample/DUP, must be extracted and analyzed. MS/MSD is extracted only if submitted by the client. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Attachment:
Attachment 01 - Mass Transitions (.doc)
Attachment 02 - Standard Relationships (.docx)
Attachment 03 - Acquisition Method (.pdf)
Attachment 04 - Example Certificate of Analysis (.pdf)
Attachment 05 – 1633 Native PFAS Intermediate A (.pdf)
Attachment 06 – 1633 Native PFAS Intermediate B (.pdf)
Attachment 07 – 1633 EIS Intermediate (.pdf)
Attachment 08 – 1633 NIS Intermediate (.pdf)
Attachment 09 – 1633 TOP SS Intermediate (.pdf)
Attachment 10 - 1633 ICAL Stds Preparation Scheme (.pdf)
Attachment 11 - 1633 Initial Calibration Standard Concentrations (.pdf)
Attachment 12 - Bile Salts Stock Solution (.pdf)
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Attachment 17 - Native Low Level Spike Intermediate (.pdf)
Attachment 18 - Native Low Level Spike (.pdf)
Attachment 19 - 1633 ICV Standard (.pdf)
Attachment 20 - 1633 Purchased Standards (.pdf)
Attachment 21 - Table 5 (1633 Draft, V3) (.docx)
Attachment 22 - Table 8 (1633 Draft, V3) (.docx)

G-DC-FRM23907 Redacted SOPs
QA-SOP11178 Demonstrations of Capability
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
T-PEST-WI9847 Common Equations Used During Chromatographic Analyses
T-PFAS-WI21568 Manifold and N-EVAP Cleaning for PFAS Extractions

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T-WC-WI53304 Total Suspended Solids (TSS Gravimetric) Prescreen for PFAS by EPA Draft Methods in Aqueous Samples
Attachment: Attachment 01 - Mass Transitions (doc)
Attachment: Attachment 02 - Standard Relationships (docx)
Attachment: Attachment 03 - Acquisition Method (pdf)
Attachment: Attachment 04 - Example Certificate of Analysis (pdf)
Attachment: Attachment 05 – 1633 Native PFAS Intermediate A (pdf)
Attachment: Attachment 06 – 1633 Native PFAS Intermediate B (pdf)
Attachment: Attachment 07 – 1633 EIS Intermediate (pdf)
Attachment: Attachment 08 – 1633 NIS Intermediate (pdf)
Attachment: Attachment 09 – 1633 TOP SS Intermediate (pdf)
Attachment: Attachment 10 - 1633 ICAL Stds Preparation Scheme (pdf)
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Attachment: Attachment 18 - Native Low Level Spike (pdf)
Attachment: Attachment 19 - 1633 ICV Standard (pdf)
Attachment: Attachment 20 - 1633 Purchased Standards (pdf)
Attachment: Attachment 21 - Table 5 (1633 Draft, V3) (docx)
Attachment: Attachment 22 - Table 8 (1633 Draft, V3) (docx)

End of document

Version history

Version	Approval	Revision information	
2	13.OCT.2022		
3	09.MAY.2023		
4	07.DEC.2023		

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Attachment 1

Mass Transitions AB Sciex 4500/5500/5500+

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216.0	172.0
13C4-PFBA	216.8	171.9
PFBA	212.8	168.9
13C5-PFPeA	268.3	223
PFPeA	263.0	219.0
PFPeA (2)	263.0	68.9
13C3-PFBS	302.1	79.9
13C3-PFBS (2)	302.1	98.9
PFBS	298.7	79.9
PFBS (2)	298.7	98.8
13C2-4:2-FTS	329.1	80.9
13C2-4:2-FTS (2)	329.1	309.0
4:2-FTS	327.1	307.0
4:2-FTS (2)	327.1	80.9
13C2-PFHxA	315.1	270.0
13C2-PFHxA (2)	315.1	119.4
13C5-PFHxA	318.0	273.0
13C5-PFHxA (2)	318.0	120.3
PFHxA	313.0	269.0
PFHxA (2)	313.0	118.9
PFPeS	349.1	79.9
PFPeS (2)	349.1	98.9
18O2-PFHxS	403.0	83.9
13C3-PFHxS	402.1	79.9
13C3-PFHxS (2)	402.1	98.9
PFHxS	398.7	79.9
PFHxS (2)	398.7	98.9
13C4-PFHpA	367.1	322.0
PFHpA	363.1	319.0
PFHpA (2)	363.1	169.0
13C2-6:2-FTS	429.1	80.9
13C2-6:2-FTS (2)	429.1	409.0
6:2-FTS	427.1	407.0
6:2-FTS (2)	427.1	80.9
PFHpS	449.0	79.9
PFHpS (2)	449.0	98.8
13C4-PFOA	417.1	172.0

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Attachment 1

Compound	Parent Ion	Daughter Ion
13C8-PFOA	421.1	376.0
PFOA	413.0	369.0
PFOA (2)	413.0	169.0
13C4-PFOS	502.8	79.9
13C4-PFOS (2)	502.8	98.9
13C8-PFOS	507.1	79.9
13C8-PFOS (2)	507.1	98.9
PFOS	498.9	79.9
PFOS (2)	498.9	98.8
13C5-PFNA	468.0	423.0
13C9-PFNA	472.1	427.0
PFNA	463.0	419.0
PFNA (2)	463.0	219.0
13C8-PFOSA	506.1	77.8
PFOSA	498.1	77.9
PFOSA (2)	498.1	478.0
PFNS	548.8	79.9
PFNS (2)	548.8	98.8
13C2-PFDA	515.1	470.1
13C6-PFDA	519.1	474.1
PFDA	512.9	469.0
PFDA (2)	512.9	219.0
13C2-8:2-FTS	529.1	80.9
13C2-8:2-FTS (2)	529.1	509.0
8:2-FTS	527.1	507.0
8:2-FTS (2)	527.1	80.8
d7-NMePFOSAE	623.2	58.9
NMePFOSAE	616.1	58.9
d3-NMePFOSA	515.0	219.0
NMEPFOSA	511.9	219.0
NMEPFOSA (2)	511.9	169.0
d3-NMeFOSAA	573.2	419.0
NMeFOSAA	570.1	419.0
NMeFOSAA (2)	570.1	483.0
d9-NEtPFOSAE	639.2	58.9
NEtPFOSAE	630.0	58.9
d5-NETPFOSA	531.1	219.0
NEtPFOSA	526.0	219.0
NEtPFOSA (2)	526.0	169.0
PFDS	599.0	79.9

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Attachment 1

Compound	Parent Ion	Daughter Ion
PFDS (2)	599.0	98.8
13C7-PFUnDA	570.0	525.1
PFUnDA	563.1	519.0
PFUnDA (2)	563.1	269.1
d5-NEtFOSAA	589.2	419.0
NEtFOSAA	584.2	419.1
NEtFOSAA (2)	584.2	526.0
13C2-PFDoDA	615.1	570.0
PFDoDA	613.1	569.0
PFDoDA (2)	613.1	319.0
PFDoS	699.1	79.9
PFDoS (2)	699.1	98.8
PFTrDA	663.0	619.0
PFTrDA (2)	663.0	168.9
13C2-PFTeDA	715.2	670.0
PFTeDA	713.1	669.0
PFTeDA (2)	713.1	168.9
13C3-HFPODA	286.9	168.9
13C3-HFPODA (2)	286.9	184.9
HFPODA	284.9	168.9
HFPODA (2)	284.9	184.9
DONA	376.9	250.9
DONA (2)	376.9	84.8
9Cl-PF3ONS	530.8	351.0
9Cl-PF3ONS (2)	532.8	353.0
11Cl-PF3OUdS	630.9	450.9
11Cl-PF3OUdS (2)	632.9	452.9
PFECA B (NFDHA)	295.0	201.0
PFECA B(NFDHA) (2)	295.0	84.9
PFECA F (PFMPA)	229.0	84.9
3:3 FTCA	241.0	177.0
3:3 FTCA (2)	241.0	117.0
PFECA A (PFMBA)	279.0	85.1
PFEESA (PES)	314.8	134.9
PFEESA (PES) (2)	314.8	82.9
5:3 FTCA	341.0	237.1
5:3 FTCA (2)	341.0	217.0
7:3 FTCA	441.0	316.9
7:3 FTCA (2)	441.0	336.9

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Attachment 2

PFAS Injection Standards/Extraction Standards/Native Compounds

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Injection Standards

Inj Std	Internal Standard/Injection Standard
I13C3-PFBA	13C3-PFBA
I13C2-PFHxA	13C2-PFHxA
I13C4-PFOA	13C4-PFOA
I13C5-PFNA	13C5-PFNA
I13C2-PFDA	13C2-PFDA
I18O2-PFHxS	18O2-PFHxS
I13C4-PFOS	13C4-PFOS

Extraction Standards

Extraction Standard	Internal Standard
E13C4-PFBA	13C3-PFBA
E13C5-PFPeA	13C2-PFHxA
E13C5-PFHxA	
E13C4-PFHpA	
E13C3-HFPO-DA	
E13C8-PFOA	13C4-PFOA
E13C9-PFNA	13C5-PFNA
E13C6-PFDA	13C2-PFDA
E13C7-PFUnA	
E13C2-PFDoA	
E13C2-PFTeDA	
E13C3-PFBS	18O2-PFHxS
E13C3-PFHxS	
E13C2-4:2-FTS	
E13C2-6:2-FTS	
E13C2-8:2-FTS	

Extraction Standard	Internal Standard
E13C8-PFOS	13C4-PFOS
E13C8-PFOSA	
Ed3-NMeFOSA	
Ed5-NEtFOSA	
Ed3-NMeFOSAA	
Ed5-NEtFOSAA	
Ed7-NMeFOSE	
Ed9-NEtFOSE	

Native PFAS Compounds

Native	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	

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Native	Extraction Standard
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

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Acquisition Method

EPA1633_DOD_23JUL06

Mass Spec 12.500 min
Period 12.500 min
-MRM
Integrated Valve
Sciex LC System
Equilibrate
Injection

Mass Spectrometer Method Properties

Period 1:

Scans in Period: 1250
Min. Dwell Time: 3 ms
Max. Dwell Time: 250 ms
Relative Start Time: 0.00 msec
Scheduled Ionization: Off
Experiments in Period: 1
Use target Cycle Time: No
Target Cycle Time: N/A

Period 1 Experiment 1:

Scan Type: MRM (MRM)
Scheduled MRM: Yes
Polarity: Negative
Scan Mode: N/A
Ion Source: Turbo Spray
sMRM Q1Q3 Resolution: No
MRM detection window: 60 sec
Target Scan Time: 0.6000 sec
Resolution Q1: Unit
Resolution Q3: Unit
Intensity Thres.: 0.00 cps
Settling Time: 0.0000 msec
MR Pause: 5.0070 msec
MCA: No
Step Size: 0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	2.92	DP	-40.00	-40.00	13C3-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.800	171.900	2.92	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.300	223.000	3.41	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
266.000	222.000	3.41	DP	-40.00	-40.00	13C3-PFPeA
			CE	-14.00	-14.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	79.900	3.81	DP	-120.00	-120.00	13C3-PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	80.900	3.60	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	270.000	3.71	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	273.000	3.71	DP	-40.00	-40.00	13C5-PFHxA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	168.900	3.82	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
367.100	322.000	3.97	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	79.900	4.35	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
359.000	294.000	4.60	DP	-40.00	-40.00	13C2-6:2 FTUCA
			CE	-25.00	-25.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
379.000	294.000	4.60	DP	-30.00	-30.00	13C2-6:2 FTCA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	80.900	4.08	DP	-100.00	-100.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
415.000	370.000	4.20	DP	-50.00	-50.00	13C2-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.100	172.000	4.20	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.100	376.000	4.20	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	79.900	4.93	DP	-100.00	-100.00	13C4-PFOS
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	79.900	4.93	DP	-100.00	-100.00	13C8-PFOS
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
472.100	427.000	4.43	DP	-50.00	-50.00	13C9-PFNA
			CE	-18.00	-18.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
459.000	394.000	5.13	DP	-50.00	-50.00	13C2-8:2 FTUCA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
479.000	394.000	5.13	DP	-35.00	-35.00	13C2-8:2 FTCA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
519.100	474.100	4.70	DP	-50.00	-50.00	13C6-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.100	470.100	4.70	DP	-50.00	-50.00	13C2-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	80.900	4.53	DP	-100.00	-100.00	13C2-8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
506.100	77.800	5.78	DP	-100.00	-100.00	13C8-PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
573.200	419.000	4.64	DP	-80.00	-80.00	d3-NMeFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
565.000	520.000	5.07	DP	-70.00	-70.00	13C2-PFUnDA
			CE	-19.00	-19.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	525.100	5.07	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
589.200	419.000	4.78	DP	-90.00	-90.00	d5-NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.100	570.000	5.55	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.200	58.900	6.62	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	6.76	DP	-100.00	-100.00	d3-NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.200	58.900	6.90	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.100	219.000	7.13	DP	-100.00	-100.00	d5-NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.200	670.000	6.40	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

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```
212.800 168.900 2.92    DP      -40.00 -40.00 PFBA
                        CE      -14.00 -14.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param Start  Stop  ID
249.000       99.000        3.12      DP    -60.00 -60.00    PFPrS
                                      CE    -40.00-40.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param Start  Stop  ID
229.000       84.900        3.18      DP    -40.00 -40.00    PFECA F
                                      CE    -25.00-25.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param Start  Stop  ID
241.000       177.000       3.16      DP    -60.00 -60.00    3:3 FTCA
                                      CE    -12.00-12.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param Start  Stop  ID
263.000       219.000       3.41      DP    -40.00 -40.00    PFPeA
                                      CE    -14.00-14.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param Start  Stop  ID
298.700       79.900        3.80      DP    -120.00-120.00 PFBS
                                      CE    -65.00-65.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param Start  Stop  ID
279.000       85.100        3.51      DP    -40.00 -40.00    PFECA A
                                      CE    -20.00-20.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param Start  Stop  ID
314.800       134.900       3.95      DP    -60.00 -60.00  PFEESA
                                      CE    -30.00-30.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param Start  Stop  ID
295.000       201.000       3.68      DP    -70.00 -70.00    PFECA B
```

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				CE	-25.00	-25.00

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	307.000	3.60	DP	-100.00	-100.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	3.71	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	79.900	4.09	DP	-90.00	-90.00	PFPeS
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	168.900	3.82	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	319.000	3.95	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	79.900	4.35	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	250.900	4.04	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.100	3.75	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
461.000	381.000	4.80	DP	-70.00	-70.00	PFECHS
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	407.000	4.07	DP	-100.00	-100.00	6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	79.900	4.60	DP	-100.00	-100.00	PFHpS
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	4.20	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	79.900	4.93	DP	-100.00	-100.00	PFOS
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	4.44	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	316.900	4.23	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
530.800	351.000	5.28	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	79.900	5.39	DP	-100.00	-100.00	PFNS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	469.000	4.71	DP	-50.00	-50.00	PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	507.000	4.53	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	77.900	5.81	DP	-100.00	-100.00	PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	419.000	4.64	DP	-80.00	-80.00	NMeFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	79.900	5.86	DP	-100.00	-100.00	PFDS
			CE	-120.00	-120.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	519.000	5.08	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	419.100	4.77	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.900	450.900	6.18	DP	-100.00	-100.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	569.000	5.55	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.100	58.900	6.64	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	219.000	6.76	DP	-100.00	-100.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	79.900	6.62	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	58.900	6.93	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.16	DP	-100.00	-100.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	619.000	6.01	DP	-60.00	-60.00	PFTrDA
			CE	-21.00	-21.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	669.000	6.40	DP	-60.00	-60.00	PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	98.800	3.80	DP	-100.00	-100.00	PFBS_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	84.900	3.68	DP	-30.00	-30.00	PFECA B_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	80.900	3.60	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	118.900	3.71	DP	-50.00	-50.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	98.900	4.09	DP	-100.00	-100.00	PFPeS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	184.900	3.82	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
385.000	185.000	3.82	DP	-75.00	-75.00	HFPODA_3
			CE	-10.00	-10.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	169.000	3.95	DP	-60.00	-60.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	98.900	4.35	DP	-100.00	-100.00	PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	3.75	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
461.000	99.000	4.80	DP	-60.00	-60.00	PFECHS_2
			CE	-60.00	-60.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	80.900	4.07	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	98.800	4.60	DP	-100.00	-100.00	PFHpS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	4.20	DP	-60.00	-60.00	PFOA_2
			CE	-26.00	-26.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	98.800	4.93	DP	-100.00	-100.00	PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

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```
463.000 219.000 4.44    DP     -60.00 -60.00 PFNA_2
                        CE     -30.00 -30.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
548.800       98.800        5.39      DP     -100.00-100.00 PFNS_2
                                      CE     -90.00-90.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
512.900       219.000       4.71      DP     -50.00 -50.00  PFDA_2
                                      CE     -31.00-31.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
527.100       80.800        4.53      DP     -100.00-100.00   8:2 FTS_2
                                      CE     -80.00-80.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
570.100       483.000       4.64      DP     -80.00 -80.00    NMeFOSAA_2
                                      CE     -24.00-24.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
599.000       98.800        5.86      DP     -100.00-100.00   PFDS_2
                                      CE     -100.00  -100.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
563.100       269.100       5.08      DP     -80.00 -80.00    PFUnDA_2
                                      CE     -35.00-35.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
584.200       526.000       4.77      DP     -100.00-100.00   NEtFOSAA_2
                                      CE     -30.00-30.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
613.100       319.000       5.55      DP     -60.00 -60.00    PFDoDA_2
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			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	168.900	6.01	DP	-60.00	-60.00	PFTrDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	168.900	6.40	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	4.42	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	83.900	4.33	DP	-100.00	-100.00	18O2-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	68.900	3.41	DP	-60.00	-60.00	PFPeA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	478.000	5.81	DP	-130.00	-130.00	PFOSA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	169.000	6.76	DP	-100.00	-100.00	NMePFOSA_2
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.16	DP	-180.00	-180.00	NEtPFOSA_2
			CE	-40.00	-40.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	84.800	4.04	DP	-40.00	-40.00	DONA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
532.800	353.000	5.28	DP	-100.00	-100.00	9Cl-PF3ONS_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
632.900	452.900	6.18	DP	-180.00	-180.00	11Cl-PF3OUdS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	3.16	DP	-50.00	-50.00	3:3 FTCA_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	336.900	4.23	DP	-80.00	-80.00	7:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	82.900	3.95	DP	-60.00	-60.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	98.800	6.62	DP	-100.00	-100.00	PFDoS_2
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.300	3.71	DP	-180.00	-180.00	13C5-PFHxA_2
			CE	-40.00	-40.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	98.900	3.81	DP	-120.00	-120.00	13C3-PFBS_2
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	98.800	4.35	DP	-100.00	-100.00	13C3-PFHxS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	98.900	4.93	DP	-100.00	-100.00	13C8-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	309.000	3.60	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	409.000	4.08	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	509.000	4.53	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	184.900	3.82	DP	-20.00	-20.00	13C3-HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	119.400	3.71	DP	-30.00	-30.00	13C2-PFHxA_2
			CE	-18.00	-18.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	98.900	4.93	DP	-100.00	-100.00	13C4-PFOS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	80.000	3.62	DP	-100.00	-100.00	TDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	80.000	3.86	DP	-100.00	-100.00	TCDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.200	80.000	3.94	DP	-100.00	-100.00	TUDCA
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
CAD:	10.00
IS:	-3000.00
TEM:	450.00
GS1:	40.00
GS2:	50.00
EP	-10.00
CXP	-14.00

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CERTIFICATE OF ANALYSIS
DOCUMENTATION

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PFAC-MXC

Native Perfluorinated Compound Solution/Mixture

PRODUCT CODE:	PFAC-MXC
LOT NUMBER:	PFACMXC0617
SOLVENT(S):	Methanol / Water (<1%)
DATE PREPARED: (mm/ddlyyyy)	06/14/2017
LAST TESTED: (mm/dd/yyyy)	03/19/2019
EXPIRY DATE: (mmldd/yyyy)	03/19/2024
RECOMMENDED STORAGE:	Store ampoule in a cool, dark place

DESCRIPTION:

PFAC-MXC is a solution/mixture of thirteen native perfluoroalkylcarboxylic acids (C_4-C_{14}, C_6 and C,) and eight native perfluoroalkylsulfonates (C_4-C and C,J The full name, abbreviation and concentration for each of the components are given in Table A.

The individual perfluoroalkylcarboxylic acids and perfluoroalkylsulfonates all have chemical purities of >98%.

DOCUMENTATION/ DATA ATTACHED:

Table A: Components and Concentrations of the Solution/Mixture
Figure 1: LC/MS Data (SIR)
Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

See page 2 for further details.
Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acids to their respective methyl esters.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Wellington Laboratories Inc., 345 Southgate Dr. Guelph ON N1G 3M5 CANADA
519-822-2436 • Fax: 519-822-2849 • info@well-labs.com

Attachment"" to "US Eurofins US Lancaster Laboratories Environmental -Analysis of Per and Polyfiuoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633"
Printed by Erin Shortes, d. Fri 08 Mar 2024 11:27 EST

ATTACHMENT 4

INTENDED USE:

The products prepared by Wellington Laboratories Inc. are for laboratory use only. This certified reference material (CRM) was designed to be used as a standard for the identification and/or quantification of the specific chemical compounds it contains.

HANDLING:

This product should only be used by qualified personnel familiar with its potential hazards and trained in the handling of hazardous chemicals. Due care should be exercised to prevent unnecessary human contact or ingestion. All procedures should be carried out in a well-functioning fume hood and suitable gloves, eye protection, and clothing should be worn at all times. Waste should be disposed of according to national and regional regulations. Safety Data Sheets (SDSs) are available upon request.

SYNTHESIS / CHARACTERIZATION:

Our products are synthesized using single-product unambiguous routes whenever possible. They are then characterized, and their structures and purities confirmed, using a combination of the most relevant techniques, such as NMR, GC/MS, LC/MS/MS, SFC/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This further confirms the homogeneity of the crystalline material as well as the stability and homogeneity of the solutions in the storage containers. In order to maintain the integrity of the assigned value(s), and associated uncertainty, the dilution or injection of a subsample of this product should be performed using calibrated measuring equipment.

UNCERTAINTY:

The maximum combined relative standard uncertainty of our reference standard solutions is calculated using the following equation:

The combined relative standard uncertainty, uc(y), of a value *y* and the uncertainty of the independent parameters $x_1, x_2, \ldots x_n$ on which it depends is:

$$ur(y(x_i,x_1, \ldots x_n)) = \sqrt{\sum_{i=1} u(y,x_i)^2}$$

where x is expressed as a relative standard uncertainty of the individual parameter.

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of ±5% (calculated with a coverage factor of 2 and a level of confidence of 95%) is stated on the Certificate of Analysis for all of our products.

TRACEABILITY:

All reference standard solutions are traceable to specific crystalline lots. The microbalances used for solution preparation are regularly calibrated by an external ISO/IEC 17025 accredited laboratory. In addition, their calibration is verified prior to each weighing using calibrated external weights traceable to an ISO/IEC 17025 accredited laboratory. All volumetric glassware used is calibrated, of Class **A** tolerance, and traceable to an ISO/IEC 17025 accredited laboratory. For certain products, traceability to international interlaboratory studies has also been established.

EXPIRY DATE / PERIOD OF VALIDITY:

Ongoing stability studies of this product have demonstrated stability in its composition and concentration, until the specified expiry date, in the unopened ampoule. Monitoring for any degradation or change in concentration of the listed analyte(s) is performed on a routine basis.

I.JMITED WARRANTY:

At the time of shipment, all products are warranted to be free of defects in material and workmanship and to conform to the stated technical and purity specifications.

QUALITY MANAGEMENT:

This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAi Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A 1226), and ISO 17034 by ANSI-ASQ National Accreditation Board **(ANAB;** AR-1523).

CALA

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REFERENCE MATERIAL PRODUCER

For additional information or assistance concerning this or any other products from Wellington Laboratories Inc., please visit our website at www.well-labs.com or contact us directly at info@well-labs.com

Attachment"" to "US Eurofins US Lancaster Laboratories Environmental - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633"
Printed by Erin Shortes, d. Fri 08 Mar 2024 11:27 EST

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ATTACHMENT 4

Table A: **PFAC-MXC; Components and Concentrations (ng/ml, ± 5% in Methanol/ Water (<1%))**

Compound	Abbreviation	Concentration (ng/ml)*	Peak Assignment in Figure 1
Perfluoro-n-butanoic acid	PFBA	2000	A
Perfluoro-n-pentanoic acid	PFPeA	2000	B
Perfluoro-n-hexanoic acid	PFHxA	2000	D
Perfluoro-n-heptanoic acid	PFHpA	2000	F
Perfluoro-n-octanoic acid	PFOA	2000	**H**
Perfluoro-n-nonanoic acid	PFNA	2000	J
Perfluoro-n-decanoic acid	PFDA	2000	L
Perfluoro-n-undecanoic acid	PFUdA	2000	**N**
Perfluoro-n-dodecanoic acid	PFDoA	2000	P
Perfluoro-n-tridecanoic acid	PFTrDA	2000	Q
Perfluoro-n-tetradecanoic acid	PFTeDA	2000	**S**
Perfluoro-n-hexadecanoic acid	PFHxDA	2000	**T**
Perfluoro-n-octadecanoic acid	PFODA	2000	**U**

Compound	Abbreviation	Concentration (ng/ml)*		Peak Assignment in Figure 1
		As the salt	As the anion	
Potassium perfluoro-1-butanesulfonate	L-PFBS	2000	1770	C
Sodium perfluoro-1-pentanesulfonate	L-PFPeS	2000	1880	E
Sodium perfluoro-1-hexanesulfonate	L-PFHxS	2000	1890	G
Sodium perfluoro-1-heptanesulfonate	L-PFHpS	2000	1900	I
Sodium perfluoro-1-octanesulfonate	L-PFOS	2000	1910	K
Sodium perfluoro-1-nonanesulfonate	L-PFNS	2000	1920	**M**
Sodium perfluoro-1-decanesulfonate	L-PFDS	2000	1930	O
Sodium perfluoro-1-dodecanesulfonate	L-PFDoS	2000	1940	R

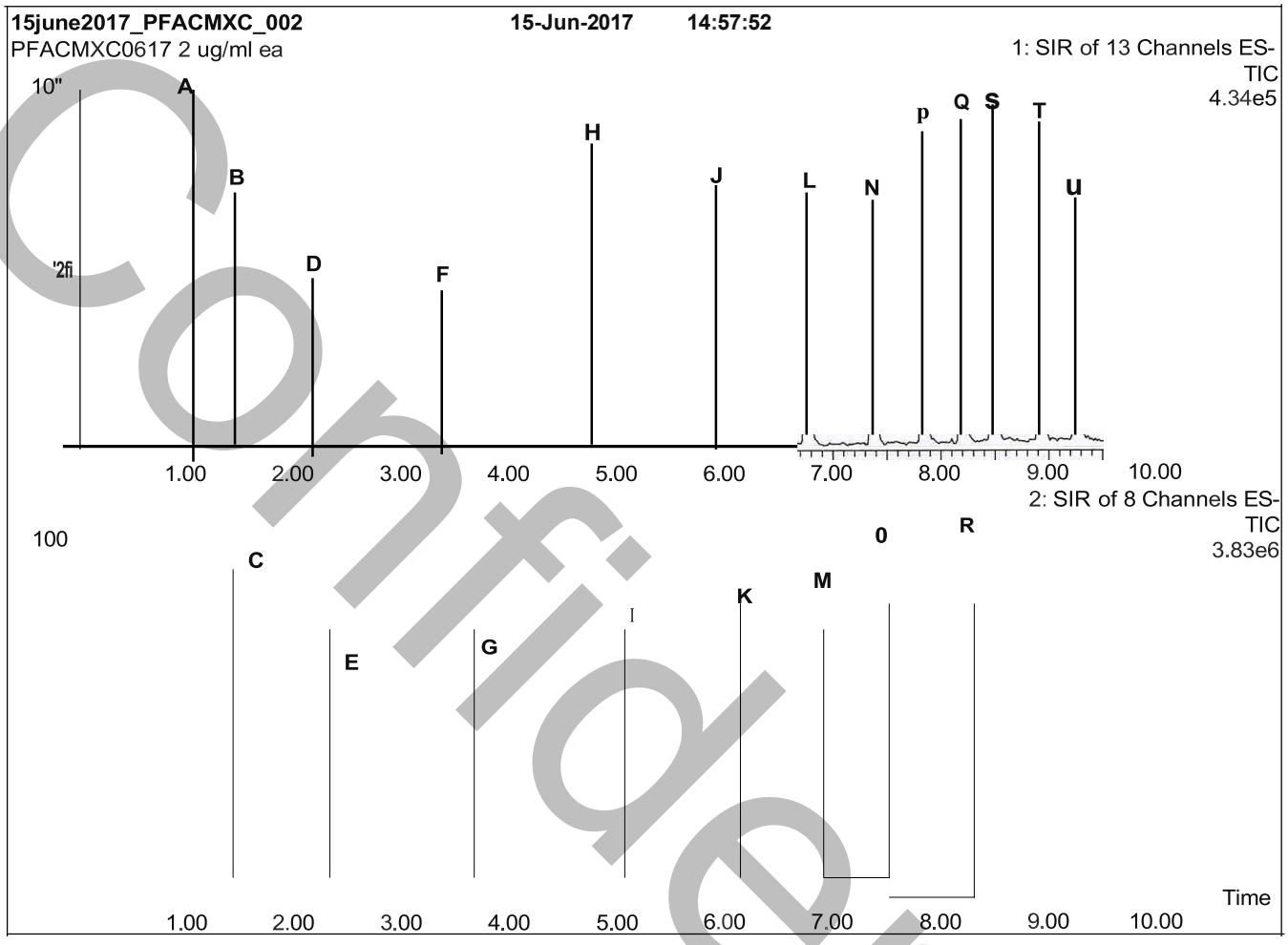
* Concentrations have been rounded to three significant figures.

Certified By: _____ **Date:** 06/06/2019
(mm/dd/yyyy)

B.G. Chittim, General Manager

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Fi.9yr!L1 **PFAC-MXC; LC/MS Data (Total Ion Current Chromatogram; SIR)**



Conditions for Figure 1:

LC: **Waters Acquity Ultra Performance LC**
MS: **Micromass Quattro *micro* API MS**

Chromatographic Conditions

Column: Acquity UPLC BEH Shield RP.,
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 50% H,O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH,OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions in 1 min.

Time: 12 min

Flow: 300 µl/min

MS Parameters

Experiment: SIR of 21 Channels

Source: Electrospray (negative)
Capillary Voltage (kV) = 3.00
Cone Voltage (V) = variable (10-80)
Cone Gas Flow (l/hr) = 50
Desolvation Gas Flow (l/hr) = 750

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Figure 2: **PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)**

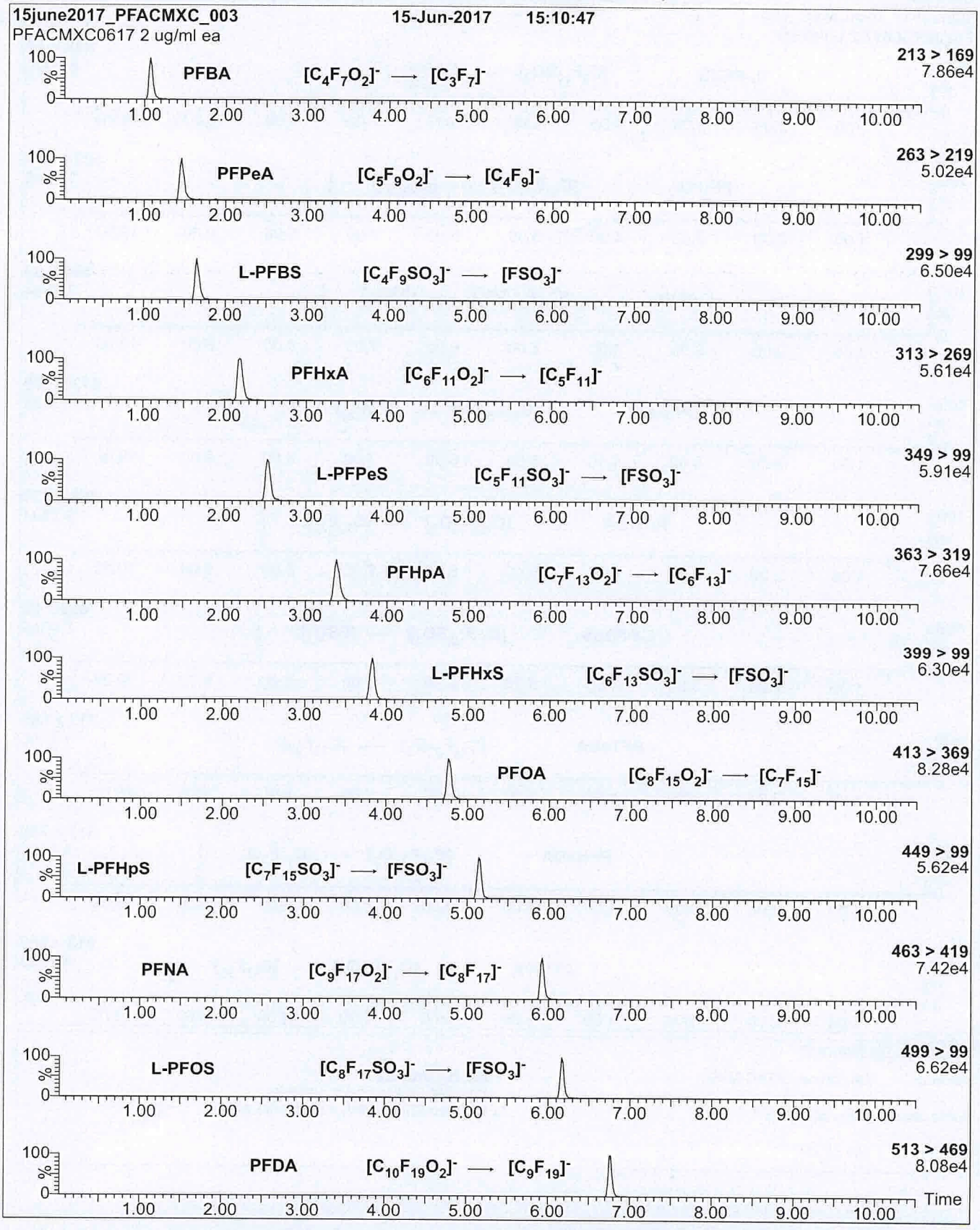
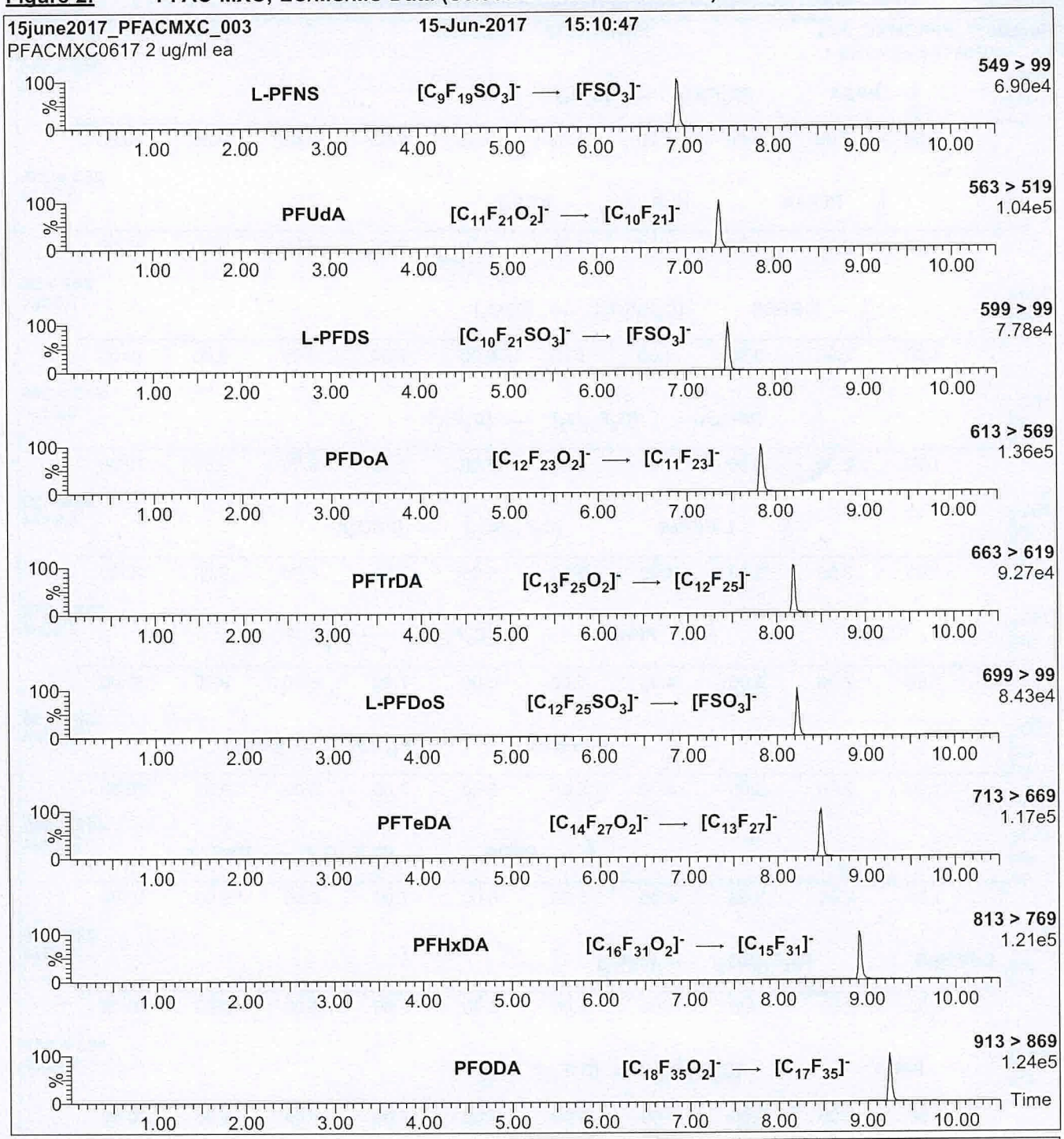


Figure 2: **PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)**



Conditions for Figure 2:

Injection: On-column (PFAC-MXC)

Mobile phase: Same as Figure 1

Flow: 300 µl/min

MS Parameters
Collision Gas (mbar) = 3.46e-3
Collision Energy (eV) = 8-50 (variable)

Native PFAS Intermediate A								
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/mL)	Aliquot (mL)	Final Volume (mL MeOH)	Final Conc. Native PFAS Intermediate (ng/ml)
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.100		471.0
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.100		466.0
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	47100	0.100		471.0
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	50000	0.100		500.0
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	47900	0.100		479.0
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	46700	0.100		467.0
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	47400	0.100		474.0
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.025		125.0
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.025		125.0
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.025		110.5
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.100		500.0
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.025		120.5
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.025		125.0
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	48400	0.025		121.0
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	50000	0.025		125.0
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.025		119.0
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.025		125.0
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.025		113.8
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.025	10	125.0
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.025		120.0
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.025		125.0
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.025		125.0
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46400	0.025		116.0
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.025		125.0
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900	0.025		117.3
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.050		250.0
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.025		125.0
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.025		125.0
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	50000	0.025		125.0
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.050		250.0
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	50000	0.050		250.0
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50000	0.050		250.0
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.050		222.5
Wellington	br-NMeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSE	50000	0.250		1250.0
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	50000	0.025		125.0
Wellington	br-NEtFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSE	50000	0.250		1250.0
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	50000	0.025		125.0
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.125		625.0
Wellington	FPePA	3-Perflurorpentylpropanoic acid	914637-49-3	5:3 FTCA	50000	0.625		3125.0
Wellington	FHpPA	3-Perfluoroheptylpropanoic acid	812-70-4	7:3 FTCA	50000	0.625		3125.0

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Native PFAS Intermediate B								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Native PFAS Intermediate (ng/ml)**
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.200		942.0
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.200		932.0
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	47100	0.200		942.0
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	50000	0.200		1000.0
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	47900	0.200		958.0
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	46700	0.200		934.0
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	47400	0.200		948.0
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.050		250.0
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.050		250.0
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.050		221.0
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.200		1000.0
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.050		241.0
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.050		250.0
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	48400	0.050		242.0
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	50000	0.050		250.0
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.050		238.0
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.050		250.0
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.050	10	227.5
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.050		250.0
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.050		240.0
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.050		250.0
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.050		250.0
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46400	0.050		232.0
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.050		250.0
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900	0.050		234.5
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.100		500.0
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.050		250.0
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.050		250.0
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	50000	0.050		250.0
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.100		500.0
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	50000	0.100		500.0
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50000	0.100		500.0
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.100		445.0
Wellington	br-NMeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	50000	0.500		2500.0
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	50000	0.050		250.0
Wellington	br-NEtFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	50000	0.500		2500.0
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	50000	0.050		250.0
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.25		1250.0

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1633 Extracted-Internal Standard (EIS) Intermediate								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL of MeOH)**	**Final Conc. Native PFAS Intermediate (ng/ml)**
Wellington	MPFBA	Perfluoro-n-($^{13}C_4$)butanoic acid	STL00992	$^{13}C_4$-PFBA	50000	0.4	10	2000.0
Wellington	M5PFPeA	Perfluoro-n-($^{13}C_5$)pentanoic acid	STL01893	$^{13}C_5$-PFBA	50000	0.2		1000.0
Wellington	M5PFHxA	Perfluoro-n-(1,2,3,4,6-$^{13}C_5$)hexanoic acid	STL02577	$^{13}C_5$-PFHxA	50000	0.1		500.0
Wellington	M4PFHpA	Perfluoro-n-(1,2,3,4-$^{13}C_4$)heptanoic acid	STL01892	$^{13}C_4$-PFHpA	50000	0.1		500.0
Wellington	M8PFOA	Perfluoro-n-($^{13}C_8$)octanoic acid	STL01052	$^{13}C_8$-PFOA	50000	0.1		500.0
Wellington	M9PFNA	Perfluoro-n-($^{13}C_9$)nonanoic acid	STL02578	$^{13}C_9$-PFNA	50000	0.05		250.0
Wellington	M6PFDA	Perfluoro-n-(1,2,3,4,5,6-$^{13}C_6$)decanoic acid	STL02579	$^{13}C_6$-PFDA	50000	0.05		250.0
Wellington	M7PFUdA	Perfluoro-n-(1,2,3,4,5,6,7-$^{13}C_7$)undecanoic acid	STL02580	$^{13}C_7$-PFUnA	50000	0.05		250.0
Wellington	MPFDoA	Perfluoro-n-(1,2-$^{13}C_2$)dodecanoic acid	STL02703	$^{13}C_2$-PFDoDA	50000	0.05		250.0
Wellington	M2PFTeDA	Perfluoro-n-(1,2-$^{13}C_2$)tetradecanoic acid	STL02116	$^{13}C_2$-PFTeDA	50000	0.05		250.0
Wellington	M3PFBS	Perfluoro-1-(2,3,4-$^{13}C_3$)butanesulfonic acid	STL02337	$^{13}C_3$-PFBS	46500	0.1		465.0
Wellington	M3PFHxS	Perfluoro-1-(1,2,3-$^{13}C_3$)hexanesulfonic acid	STL02581	$^{13}C_3$-PFHxS	47300	0.1		473.0
Wellington	M8PFOS	Perfluoro-1-($^{13}C_8$)octanesulfonic acid	STL01054	$^{13}C_8$-PFOS	47800	0.1		478.0
Wellington	M8FOSA	Perfluoro-1-($^{13}C_8$)octanesulfonamide	STL01056	$^{13}C_8$-FOSA	50000	0.1		500.0
Wellington	d3-N-MeFOSAA	N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	d_3-NMeFOSAA	50000	0.2		1000.0
Wellington	d5-N-EtFOSAA	N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	d_5-NEtFOSAA	50000	0.2		1000.0
Wellington	M2-4:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)hexanesulfonic Acid	STL02395	$^{13}C_2$-4:2FTS	46700	0.2		934.0
Wellington	M2-6:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)octanesulfonic Acid	STL02279	$^{13}C_2$-6:2FTS	47500	0.2		950.0
Wellington	M2-8:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)decanesulfonic Acid	STL02280	$^{13}C_2$-8:2FTS	47900	0.2		958.0
Wellington	M3HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)($^{13}C_3$)propanoic acid	STL02255	$^{13}C_3$-HFPO-DA	50000	0.4		2000.0
Wellington	d7-N-MeFOSE	2-(N-methyl-d_3-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02277	d_7-NMeFOSE	50000	1		5000.0
Wellington	d9-N-EtFOSE	2-(N-ethyl-d_5-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02278	d_9-NEtFOSE	50000	1		5000.0
Wellington	d-N-MeFOSA	N-ethyl-d_5-perfluoro-1-octanesulfonamide	STL02704	d_5-NEtFOSA	50000	0.1		500.0
Wellington	d-N-EtFOSA	N-methyl-d_3-perfluoro-1-octanesulfonamide	STL02705	d_3-NMeFOSA	50000	0.1		500.0

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1633 Non-extracted Internal Standard (NIS) Intermediate								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Native PFAS Intermediate (ng/ml)**
Wellington	M3PFBA	Perfluoro-n-(2,3,4-$^{13}C_3$)butanoic acid	STL02680	$^{13}C_3$-PFBA	50000	0.20	10	1000.0
Wellington	MPFOA	Perfluoro-n-(1,2,3,4-$^{13}C_4$)octanoic acid	STL00990	$^{13}C_4$-PFOA	50000	0.10		500.0
Wellington	MPFDA	Perfluoro-n-(1,2-$^{13}C_2$)decanoic acid	STL00996	$^{13}C_2$-PFDA	50000	0.05		250.0
Wellington	MPFOS	Perfluoro-1-(1,2,3,4-$^{13}C_4$)octanesulfonic Acid	STL00991	$^{13}C_4$-PFOS	47800	0.10		478.0
Wellington	MPFNA	Perfluoro-n-(1,2,3,4,5-$^{13}C_5$)nonanoic acid	STL00995	$^{13}C_5$-PFNA	50000	0.05		250.0
Wellington	MPFHxA	Perfluoro-n-(1,2-$^{13}C_2$)hexanoic acid	STL00993	$^{13}C_2$-PFHxA	50000	0.10		500.0
Wellington	MPFHxS	Perfluoro-1-hexane($^{18}O_2$)sulfonic acid	STL00994	$^{18}O_2$-PFHxS	47300	0.10		473.0

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1633 TOP SS Intermediate								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc 1633 TOP Intermediate (ng/ml)**
Wellington	M2PFOA	Perfluoro-n-(1,2-$^{13}C_2$)octanoic acid	STL00623	$^{13}C_2$-PFOA	50000	0.020	2	500
Wellington	MPFUdA	Perfluoro-n-(1,2-$^{13}C_2$)undecanoic acid	STL00997	$^{13}C_2$-PFUnA	50000	0.010		250
Wellington	M3PFPeA	Perfluoro-n-(3,4,5-$^{13}C_3$)pentanoic acid	STL03269	^{13}C3-PFPeA	50000	0.040		1000

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1633 Initial Calibration Standards Preparation							
Solution Name	**CAL1**	**CAL2**	**CAL3**	**CAL4**	**CAL5**	**CAL6**	**CAL7**
Native PFAS Intermediate A (mL)	0.016	0.040	0.100	0.200	0.400	NA	NA
Native PFAS Intermediate B (mL)	NA	NA	NA	NA	NA	0.500	2.500
1633 TOP SS Intermediate (mL)	0.050	0.050	0.050	0.050	0.050	0.050	0.050
1633 EIS Intermediate (mL)	0.050	0.050	0.050	0.050	0.050	0.050	0.050
1633 NIS Intermediate (mL)	0.050	0.050	0.050	0.050	0.050	0.050	0.050
Native FPePA Stock (5:3 FTCA) (mL)	NA	NA	NA	NA	NA	0.062	0.312
Native FHpPA Stock (7:3 FTCA) (mL)	NA	NA	NA	NA	NA	0.062	0.312
Final Volume (mL) (methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid)	10	10	10	10	10	10	10

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1633 Initial Calibration Standards Concentrations							
	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7
Compound Name	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)
PFBA	0.8	2	5	10	20	50	250
PFPeA	0.4	1	2.5	5	10	25	125
PFHxA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFHpA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFOA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFNA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFUnA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDoA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFTrDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFTeDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFBS	0.1768	0.442	1.105	2.21	4.42	11.05	55.25
PFPeS	0.1876	0.469	1.1725	2.345	4.69	11.725	58.625
PFHxS	0.182	0.455	1.1375	2.275	4.55	11.375	56.875
PFHpS	0.1904	0.476	1.19	2.38	4.76	11.05	55.25
PFOS	0.1856	0.464	1.16	2.32	4.64	11.6	58
PFNS	0.192	0.48	1.2	2.4	4.8	12	60
PFDS	0.1928	0.482	1.205	2.41	4.82	12.05	60.25
PFDoS	0.1936	0.484	1.21	2.42	4.84	12.1	60.5
8:2FTS	0.7664	1.916	4.79	9.58	19.16	47.9	239.5
4:2FTS	0.7472	1.868	4.67	9.34	18.68	46.7	233.5
6:2FTS	0.7584	1.896	4.74	9.48	18.96	47.4	237
PFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NEtFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSAA	0.2	0.5	1.25	2.5	5	12.5	62.5
NEtFOSAA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSE	2	5	12.5	25	50	125	625
NEtFOSE	2	5	12.5	25	50	125	625
HFPO-DA	0.8	2	5	10	20	50	250
ADONA	0.7536	1.884	4.71	9.42	18.84	47.1	235.5
PFMPA	0.4	1	2.5	5	10	25	125
PFMBA	0.4	1	2.5	5	10	25	125
NFDHA	0.4	1	2.5	5	10	25	125
9Cl-PF3ONS	0.7456	1.864	4.66	9.32	18.64	46.6	233
11Cl-PF3OUdS	0.7536	1.884	4.71	9.42	18.84	47.1	235.5
PFEESA	0.356	0.89	2.225	4.45	8.9	22.25	111.25
3:3FTCA	1	2.5	6.25	12.5	25	62.5	312.5
5:3FTCA	5	12.5	31.25	62.5	125	312	1560
7:3FTCA	5	12.5	31.25	62.5	125	312	1560
13C4-PFBA	10	10	10	10	10	10	10
13C5-PFPeA	5	5	5	5	5	5	5
13C5-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C4-PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C8-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C9-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C6-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C7-PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFTeDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C3-PFBS	2.33	2.33	2.33	2.33	2.33	2.33	2.33
13C3-PFHxS	2.37	2.37	2.37	2.37	2.37	2.37	2.37
13C8-PFOS	2.395	2.395	2.395	2.395	2.395	2.395	2.395
13C2-4:2 FTS	4.69	4.69	4.69	4.69	4.69	4.69	4.69
13C2-6:2 FTS	4.755	4.755	4.755	4.755	4.755	4.755	4.755
13C2-8:2 FTS	4.8	4.8	4.8	4.8	4.8	4.8	4.8
13C8-PFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D3-NMeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D5-NEtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D3-NMeFOSAA	5	5	5	5	5	5	5
D5-NEtFOSAA	5	5	5	5	5	5	5
D7-NMeFOSE	25	25	25	25	25	25	25
D9-NEtFOSE	25	25	25	25	25	25	25
13C2-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C2-PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C3-PFPeA	5	5	5	5	5	5	5
13C3-HFPO-DA	10	10	10	10	10	10	10
13C3-PFBA	5	5	5	5	5	5	5
13C2-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C4-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C5-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
18O2-PFHxS	2.37	2.37	2.37	2.37	2.37	2.37	2.37
13C4-PFOS	2.395	2.395	2.395	2.395	2.395	2.395	2.395

Controlled

Bile Salt Stock Solution							
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Aliquot (g)**	**Final Volume (mL MeOH)**	**Final Conc. Bile Salt Stock Solution Adjusted for Acid (ng/ml)**
Sigma Alrich	T0557-500MG	Taurodeoxycholic Acid (TDCA)	516-50-7	TDCA	0.05	50	1000000
Sigma Alrich	T6260-1G	Taurochenodeoxycholic Acid (TCDCA)	516-35-8	TCDCA	0.05		950000
Calbiochem	580549-1GM	Tauroursodeoxycholic Acid (TUDCA)	14605-22-2	TUDCA	0.05		1000000

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Bile Salts Intermediate							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (mL)**	**Final Volume (mL) Methanol**	**Final Conc. Bile Salt Intermediate Solution (ng/ml)**
Bile Salts Stock Solution	Taurodeoxycholic Acid (TDCA)	516-50-7	TDCA	1000000	1	4	250000
	Taurochenodeoxycholic Acid (TCDCA)	516-35-8	TCDCA	950000			237500
	Tauroursodeoxycholic Acid (TUDCA)	14605-22-2	TUDCA	1000000			250000

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Bile Salts Working Solution							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Bile Salts Working Solution (ng/ml)**
Bile Salts Intermediate	Taurodeoxycholic Acid (TDCA)	516-50-7	TDCA	250000	0.10	5	5000
	Taurochenodeoxycholic Acid (TCDCA)	516-35-8	TCDCA	237500			4750
	Tauroursodeoxycholic Acid (TUDCA)	14605-22-2	TUDCA	250000			5000

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1633 Window-Defining Matrix Standard							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume* (ml)**	**Final Conc. 1633 WDM Solution (ng/ml)**
Bile Salt Working Solution	Taurodeoxycholic Acid (TDCA)	516-50-7	TDCA	5000	0.20	10	100
	Taurochenodeoxycholic Acid (TCDCA)	516-35-8	TCDCA	4750			95
	Tauroursodeoxycholic Acid (TUDCA)	14605-22-2	TUDCA	5000			100
1633 TOP SS Intermediate	Perfluoro-n-(1,2-$^{13}C_2$)octanoic acid	STL00623	$^{13}C_2$-PFOA	500	0.05		2.500
	Perfluoro-n-(1,2-$^{13}C_2$)undecanoic acid	STL00997	$^{13}C_2$-PFUnA	250			1.250
	Perfluoro-n-(3,4,5-$^{13}C_3$)pentanoic acid	STL03269	^{13}C3-PFPeA	1000			5.000
EIS Intermediate	Perfluoro-n-($^{13}C_4$)butanoic acid	STL00992	$^{13}C_4$-PFBA	2000	0.05		10.000
	Perfluoro-n-($^{13}C_5$)pentanoic acid	STL01893	$^{13}C_5$-PFBA	1000			5.000
	Perfluoro-n-(1,2,3,4,6-$^{13}C_5$)hexanoic acid	STL02577	$^{13}C_5$-PFHxA	500			2.500
	Perfluoro-n-(1,2,3,4-$^{13}C_4$)heptanoic acid	STL01892	$^{13}C_4$-PFHpA	500			2.500
	Perfluoro-n-($^{13}C_8$)octanoic acid	STL01052	$^{13}C_8$-PFOA	500			2.500
	Perfluoro-n-($^{13}C_9$)nonanoic acid	STL02578	$^{13}C_9$-PFNA	250			1.250
	Perfluoro-n-(1,2,3,4,5,6-$^{13}C_6$)decanoic acid	STL02579	$^{13}C_6$-PFDA	250			1.250
	Perfluoro-n-(1,2,3,4,5,6,7-$^{13}C_7$)undecanoic acid	STL02580	$^{13}C_7$-PFUnA	250			1.250
	Perfluoro-n-(1,2-$^{13}C_2$)dodecanoic acid	STL02703	$^{13}C_2$-PFDoDA	250			1.250
	Perfluoro-n-(1,2-$^{13}C_2$)tetradecanoic acid	STL02116	$^{13}C_2$-PFTeDA	250			1.250
	Perfluoro-1-(2,3,4-$^{13}C_3$)butanesulfonic acid	STL02337	$^{13}C_3$-PFBS	465			2.325
	Perfluoro-1-(1,2,3-$^{13}C_3$)hexanesulfonic acid	STL02581	$^{13}C_3$-PFHxS	473			2.365
	Perfluoro-1-($^{13}C_8$)octanesulfonic acid	STL01054	$^{13}C_8$-PFOS	478			2.390
	Perfluoro-1-($^{13}C_8$)octanesulfonamide	STL01056	$^{13}C_8$-FOSA	500			2.500
	N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	d_3-NMeFOSAA	1000			5.000
	N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	d_5-NEtFOSAA	1000			5.000
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)hexanesulfonic Acid	STL02395	$^{13}C_2$-4:2FTS	934			4.670
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)octanesulfonic Acid	STL02279	$^{13}C_2$-6:2FTS	950			4.750
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)decanesulfonic Acid	STL02280	$^{13}C_2$-8:2FTS	958			4.790
	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)($^{13}C_3$)propanoic acid	STL02255	$^{13}C_3$-HFPO-DA	2000			10.000
	2-(N-methyl-d_3-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02277	d_7-NMeFOSE	5000			25.000
	2-(N-ethyl-d_5-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02278	d_9-NEtFOSE	5000			25.000
	N-ethyl-d_5-perfluoro-1-octanesulfonamide	STL02704	d_5-NEtFOSA	500			2.500
	N-methyl-d_3-perfluoro-1-octanesulfonamide	STL02705	d_3-NMeFOSA	500			2.500
NIS Intermediate	Perfluoro-n-(2,3,4-$^{13}C_3$)butanoic acid	STL02680	$^{13}C_3$-PFBA	1000	0.05		5.000
	Perfluoro-n-(1,2,3,4-$^{13}C_4$)octanoic acid	STL00990	$^{13}C_4$-PFOA	500			2.500
	Perfluoro-n-(1,2-$^{13}C_2$)decanoic acid	STL00996	$^{13}C_2$-PFDA	250			1.250
	Perfluoro-1-(1,2,3,4-$^{13}C_4$)octanesulfonic Acid	STL00991	$^{13}C_4$-PFOS	478			2.390
	Perfluoro-n-(1,2,3,4,5-$^{13}C_5$)nonanoic acid	STL00995	$^{13}C_5$-PFNA	250			1.250
	Perfluoro-n-(1,2-$^{13}C_2$)hexanoic acid	STL00993	$^{13}C_2$-PFHxA	500			2.500
	Perfluoro-1-hexane($^{18}O_2$)sulfonic acid	STL00994	$^{18}O_2$-PFHxS	473			2.365

* Bring to final volume using methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid

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Native 1633 Mid-Level Spike								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Native 1633 Mid-Level Spike (ng/ml)**
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.20		942.0
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.20		932.0
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	47100	0.20		942.0
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	50000	0.20		1000.0
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	47900	0.20		958.0
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	46700	0.20		934.0
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	47400	0.20		948.0
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.05		250.0
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.05		250.0
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.05		221.0
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.20		1000.0
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.05		241.0
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.05		250.0
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	48400	0.05		242.00
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	50000	0.05		250.00
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.05		238.00
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.05		250.00
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.05		227.50
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.05		250.00
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.05	10	240.00
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.05		250.00
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.05		250.00
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46400	0.05		232.00
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.05		250.00
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900	0.05		234.50
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.10		500.00
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.05		250.00
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.05		250.00
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	50000	0.05		250.00
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.10		500.00
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	50000	0.10		500.00
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50000	0.10		500.00
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.10		445.00
Wellington	br-NMeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSE	50000	0.50		2500.00
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	50000	0.05		250.00
Wellington	br-NEtFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSE	50000	0.50		2500.00
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	50000	0.05		250.00
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.25		1250.00
Wellington	FPePA	3-Perflurorpentylpropanoic acid	914637-49-3	5:3 FTCA	50000	1.25		6250.00
Wellington	FHpPA	3-Perfluoroheptylpropanoic acid	812-70-4	7:3 FTCA	50000	1.25		6250.00

Draft

Native 1633 Low-Level Intermediate								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Native 1633 Low-Level Spike (ng/ml)**
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.40		1884.0
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.40		1864.0
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	47100	0.40		1884.0
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	50000	0.40		2000.0
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	47900	0.40		1916.0
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	46700	0.40		1868.0
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	47400	0.40		1896.0
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.10		500.0
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.10		500.0
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.10		442.0
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.40		2000.0
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.10		482.0
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.10		500.0
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	48400	0.10		484.0
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	50000	0.10		500.0
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.10		476.0
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.10		500.0
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.10	10	455.0
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.10		500.0
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.10		480.0
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.10		500.0
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.10		500.0
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46400	0.10		464.0
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.10		500.0
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900	0.10		469.0
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.20		1000.0
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.10		500.0
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.10		500.0
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	50000	0.10		500.0
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.20		1000.0
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	50000	0.20		1000.0
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50000	0.20		1000.0
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.20		890.0
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	50000	0.10		500.0
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	50000	0.10		500.0
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.50		2500.0

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Native 1633 Low-Level Spike								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Native 1633 Low-Level Spike (ng/ml)**
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	1884			188.40
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	1864			186.40
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	1884			188.40
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	2000			200.00
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	1916			191.60
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	1868			186.80
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	1896			189.60
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	500			50.00
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	500			50.00
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	442			44.20
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	2000			200.00
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	482			48.20
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	500			50.00
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	484			48.40
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	500			50.00
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	476			47.60
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	500			50.00
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	455	1.00		45.50
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	500			50.00
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	480			48.00
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	500		10	50.00
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	500			50.00
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	464			46.40
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	500			50.00
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	469			46.90
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	1000			100.00
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	500			50.00
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	500			50.00
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	500			50.00
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	1000			100.00
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	1000			100.00
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	1000			100.00
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	890			89.00
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500			50.00
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500			50.00
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	2500			250.00
Wellington	FPePA	3-Perflurorpentylpropanoic acid	914637-49-3	5:3 FTCA	50000	0.250		1250.00
Wellington	FHpPA	3-Perfluoroheptylpropanoic acid	812-70-4	7:3 FTCA	50000	0.2500		1250.00
Wellington	N-MeFOSE-M	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSAE	50000	0.1000		500.00
Wellington	N-EtFOSE-M	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NEtPFOSAE	50000	0.1000		500.00

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PFAS 1633 ICV Working Standard							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (ml)**	**Final Volume* (ml)**	**Final Conc. PFAS 1633 ICV Working Standard (ng/ml)**
ICV Native PFAS Intermediate A	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	471.0	0.2	10	9.42
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	466.0			9.32
	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	471.0			9.42
	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	500			10.00
	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	479			9.58
	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	467.0			9.34
	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	474			9.48
	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			2.50
	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			2.50
	Perfluorobutanesulfonic acid	375-73-5	PFBS	110.5			2.21
	Perfluorobutanoic acid	375-22-4	PFBA	500			10.00
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120.5			2.41
	Perfluorodecanoic acid	335-76-2	PFDA	125			2.50
	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	121.0			2.42
	Perfluorododecanoic acid	307-55-1	PFDoDA	125			2.50
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119.0			2.38
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			2.50
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	113.8			2.28
	Perfluorohexanoic acid	307-24-4	PFHxA	125			2.50
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120.0			2.40
	Perfluorononanoic acid	375-95-1	PFNA	125			2.50
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			2.50
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	116.0			2.32
	Perfluorooctanoic acid	335-67-1	PFOA	125			2.50
	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	117.3			2.35
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			5.00
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			2.50
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			2.50
	Perfluoroundecanoic acid	2058-94-8	PFUnDA	125			2.50
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			5.00
	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	250			5.00
	Perfluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	250			5.00
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	222.5			4.45
	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSE	1250			25.00
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	125			2.50
	2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	1691-99-2	NEtPFOSE	1250			25.00
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	125			2.50
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			12.50
5:3 FTCA Stock	3-Perflurorpentylpropanoic acid	914637-49-3	5:3 FTCA	3125	0.0125		3.91
7:3 FTCA Stock	3-Perfluoroheptylpropanoic acid	812-70-4	7:3 FTCA	3125	0.0125		3.91
1633 EIS Intermediate	Perfluoro-n-($^{13}C_4$)butanoic acid	STL00992	$^{13}C_4$-PFBA	2000	0.050		10.00
	Perfluoro-n-($^{13}C_5$)pentanoic acid	STL01893	$^{13}C_5$-PFBA	1000			5.00
	Perfluoro-n-(1,2,3,4,6-$^{13}C_5$)hexanoic acid	STL02577	$^{13}C_5$-PFHxA	500			2.50
	Perfluoro-n-(1,2,3,4-$^{13}C_4$)heptanoic acid	STL01892	$^{13}C_4$-PFHpA	500			2.50
	Perfluoro-n-($^{13}C_8$)octanoic acid	STL01052	$^{13}C_8$-PFOA	500			2.50
	Perfluoro-n-($^{13}C_9$)nonanoic acid	STL02578	$^{13}C_9$-PFNA	250			1.25
	Perfluoro-n-(1,2,3,4,5,6-$^{13}C_6$)decanoic acid	STL02579	$^{13}C_6$-PFDA	250			1.25
	Perfluoro-n-(1,2,3,4,5,6,7-$^{13}C_7$)undecanoic acid	STL02580	$^{13}C_7$-PFUnA	250			1.25
	Perfluoro-n-(1,2-$^{13}C_2$)dodecanoic acid	STL02703	$^{13}C_2$-PFDoDA	250			1.25
	Perfluoro-n-(1,2-$^{13}C_2$)tetradecanoic acid	STL02116	$^{13}C_2$-PFTeDA	250			1.25
	Perfluoro-1-(2,3,4-$^{13}C_3$)butanesulfonic acid	STL02337	$^{13}C_3$-PFBS	465			2.33
	Perfluoro-1-(1,2,3-$^{13}C_3$)hexanesulfonic acid	STL02581	$^{13}C_3$-PFHxS	473			2.37
	Perfluoro-1-($^{13}C_8$)octanesulfonic acid	STL01054	$^{13}C_8$-PFOS	478			2.39
	Perfluoro-1-($^{13}C_8$)octanesulfonamide	STL01056	$^{13}C_8$-FOSA	500			2.50
	N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	d_3-NMeFOSAA	1000			5.00
	N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	d_5-NEtFOSAA	1000			5.00
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)hexanesulfonic Acid	STL02395	$^{13}C_2$-4:2FTS	934			4.67
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)octanesulfonic Acid	STL02279	$^{13}C_2$-6:2FTS	950			4.75
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)decanesulfonic Acid	STL02280	$^{13}C_2$-8:2FTS	958			4.79
	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)($^{13}C_3$)propanoic acid	STL02255	$^{13}C_3$-HFPO-DA	2000			10.00
	2-(N-methyl-d_3-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02277	d_7-NMeFOSE	5000			25.00
	2-(N-ethyl-d_5-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02278	d_9-NEtFOSE	5000			25.00
	N-ethyl-d_5-perfluoro-1-octanesulfonamide	STL02704	d_5-NEtFOSA	500			2.50
	N-methyl-d_3-perfluoro-1-octanesulfonamide	STL02705	d_3-NMeFOSA	500			2.50
1633 TOP SS Intermediate	Perfluoro-n-(1,2-$^{13}C_2$)octanoic acid	STL00623	$^{13}C_2$-PFOA	500	0.050		2.50
	Perfluoro-n-(1,2-$^{13}C_2$)undecanoic acid	STL00997	$^{13}C_2$-PFUnA	250			1.25
	Perfluoro-n-(3,4,5-$^{13}C_3$)pentanoic acid	STL03269	^{13}C3-PFPeA	1000			5.00
1633 NIS Intermediate	Perfluoro-n-(2,3,4-$^{13}C_3$)butanoic acid	STL02680	$^{13}C_3$-PFBA	1000	0.05		5.00
	Perfluoro-n-(1,2,3,4-$^{13}C_4$)octanoic acid	STL00990	$^{13}C_4$-PFOA	500			2.50
	Perfluoro-n-(1,2-$^{13}C_2$)decanoic acid	STL00996	$^{13}C_2$-PFDA	250			1.25
	Perfluoro-1-(1,2,3,4-$^{13}C_4$)octanesulfonic Acid	STL00991	$^{13}C_4$-PFOS	478			2.39
	Perfluoro-n-(1,2,3,4,5-$^{13}C_5$)nonanoic acid	STL00995	$^{13}C_5$-PFNA	250			1.25
	Perfluoro-n-(1,2-$^{13}C_2$)hexanoic acid	STL00993	$^{13}C_2$-PFHxA	500			2.50
	Perfluoro-1-hexane($^{18}O_2$)sulfonic acid	STL00994	$^{18}O_2$-PFHxS	473			2.37

* Bring to final volume using methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid

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Purchased Standards					
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/mL)
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	50000
Calbiochem	580549-1GM	Tauroursodeoxycholic Acid (TUDCA)	14605-22-2	TUDCA	neat
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50000
Wellington	br-NEtFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	1691-99-2	NEtPFOSE	50000
Wellington	br-NEtFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	1691-99-2	NEtPFOSAE	50000
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46400
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	50000
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000
Wellington	br-NMeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSE	50000
Wellington	br-NMeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSAE	50000
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	47400
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	50000
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	50000
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	47900
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	50000
Sigma Alrich	T6260-1G	Taurochenodeoxycholic Acid (TCDCA)	516-35-8	TCDCA	neat
Sigma Alrich	T0557-500MG	Taurodeoxycholic Acid (TDCA)	516-50-7	TDCA	neat
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	46700
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	48400
Wellington	FHpPA	3-Perfluoroheptylpropanoic acid	812-70-4	7:3 FTCA	50000
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	50000
Wellington	FPePA	3-Perflurorpentylpropanoic acid	914637-49-3	5:3 FTCA	50000
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	47100
Wellington	M2PFOA	Perfluoro-n-(1,2-$^{13}C_2$)octanoic acid	STL00623	$^{13}C_2$-PFOA	50000
Wellington	MPFOA	Perfluoro-n-(1,2,3,4-$^{13}C_4$)octanoic acid	STL00990	$^{13}C_4$-PFOA	50000
Wellington	MPFOS	Perfluoro-1-(1,2,3,4-$^{13}C_4$)octanesulfonic Acid	STL00991	$^{13}C_4$-PFOS	47800
Wellington	MPFBA	Perfluoro-n-($^{13}C_4$)butanoic acid	STL00992	$^{13}C_4$-PFBA	50000
Wellington	MPFHxA	Perfluoro-n-(1,2-$^{13}C_2$)hexanoic acid	STL00993	$^{13}C_2$-PFHxA	50000
Wellington	MPFHxS	Perfluoro-1-hexane($^{18}O_2$)sulfonic acid	STL00994	$^{18}O_2$-PFHxS	47300
Wellington	MPFNA	Perfluoro-n-(1,2,3,4,5-$^{13}C_5$)nonanoic acid	STL00995	$^{13}C_5$-PFNA	50000
Wellington	MPFDA	Perfluoro-n-(1,2-$^{13}C_2$)decanoic acid	STL00996	$^{13}C_2$-PFDA	50000
Wellington	MPFUdA	Perfluoro-n-(1,2-$^{13}C_2$)undecanoic acid	STL00997	$^{13}C_2$-PFUnA	50000
Wellington	M8PFOA	Perfluoro-n-($^{13}C_8$)octanoic acid	STL01052	$^{13}C_8$-PFOA	50000
Wellington	M8PFOS	Perfluoro-1-($^{13}C_8$)octanesulfonic acid	STL01054	$^{13}C_8$-PFOS	47800
Wellington	M8FOSA	Perfluoro-1-($^{13}C_8$)octanesulfonamide	STL01056	$^{13}C_8$-FOSA	50000
Wellington	M4PFHpA	Perfluoro-n-(1,2,3,4-$^{13}C_4$)heptanoic acid	STL01892	$^{13}C_4$-PFHpA	50000
Wellington	M5PFPeA	Perfluoro-n-($^{13}C_5$)pentanoic acid	STL01893	$^{13}C_5$-PFBA	50000
Wellington	M2PFTeDA	Perfluoro-n-(1,2-$^{13}C_2$)tetradecanoic acid	STL02116	$^{13}C_2$-PFTeDA	50000
Wellington	d5-N-EtFOSAA	N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	d_5-NEtFOSAA	50000
Wellington	d3-N-MeFOSAA	N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	d_3-NMeFOSAA	50000
Wellington	M3HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)($^{13}C_3$)propanoic acid	STL02255	$^{13}C_3$-HFPO-DA	50000
Wellington	d7-N-MeFOSE	2-(N-methyl-d_3-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02277	d_7-NMeFOSE	50000
Wellington	d9-N-EtFOSE	2-(N-ethyl-d_5-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02278	d_9-NEtFOSE	50000
Wellington	M2-6:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)octanesulfonic Acid	STL02279	$^{13}C_2$-6:2FTS	47500
Wellington	M2-8:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)decanesulfonic Acid	STL02280	$^{13}C_2$-8:2FTS	47900
Wellington	M3PFBS	Perfluoro-1-(2,3,4-$^{13}C_3$)butanesulfonic acid	STL02337	$^{13}C_3$-PFBS	46500
Wellington	M2-4:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)hexanesulfonic Acid	STL02395	$^{13}C_2$-4:2FTS	46700
Wellington	M5PFHxA	Perfluoro-n-(1,2,3,4,6-$^{13}C_5$)hexanoic acid	STL02577	$^{13}C_5$-PFHxA	50000
Wellington	M9PFNA	Perfluoro-n-($^{13}C_9$)nonanoic acid	STL02578	$^{13}C_9$-PFNA	50000
Wellington	M6PFDA	Perfluoro-n-(1,2,3,4,5,6-$^{13}C_6$)decanoic acid	STL02579	$^{13}C_6$-PFDA	50000
Wellington	M7PFUdA	Perfluoro-n-(1,2,3,4,5,6,7-$^{13}C_7$)undecanoic acid	STL02580	$^{13}C_7$-PFUnA	50000
Wellington	M3PFHxS	Perfluoro-1-(1,2,3-$^{13}C_3$)hexanesulfonic acid	STL02581	$^{13}C_3$-PFHxS	47300
Wellington	M3PFBA	Perfluoro-n-(2,3,4-$^{13}C_3$)butanoic acid	STL02680	$^{13}C_3$-PFBA	50000
Wellington	MPFDoA	Perfluoro-n-(1,2-$^{13}C_2$)dodecanoic acid	STL02703	$^{13}C_2$-PFDoDA	50000
Wellington	d-N-MeFOSA	N-ethyl-d_5-perfluoro-1-octanesulfonamide	STL02704	d_5-NEtFOSA	50000
Wellington	d-N-EtFOSA	N-methyl-d_3-perfluoro-1-octanesulfonamide	STL02705	d_3-NMeFOSA	50000
Wellington	M3PFPeA	Perfluoro-n-(3,4,5-$^{13}C_3$)pentanoic acid	STL03269	$^{13}C3$-PFPeA	50000

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Table 5. IPR/OPR/LLOPR Acceptance Limits for Wastewater Samples

	Aqueous Matrix [1, 2]			
	IPR			
Compounds	Recovery (%)	RSD (%)	OPR Recovery (%)	LLOPR Recovery (%)
PFBA	60 – 147	20	58 – 148	44 - 157
PFPeA	56 – 150	20	54 – 152	57 - 148
PFHxA	59 – 148	25	55 – 152	62 - 149
PFHpA	60 – 149	25	54 – 154	56 - 150
PFOA	55 – 158	25	52 – 161	57 - 161
PFNA	64 – 144	25	59 – 149	53 - 157
PFDA	57 – 142	25	52 – 147	43 - 158
PFUnA	54 – 153	30	48 – 159	50 - 155
PFDoA	73 – 133	25	64 – 142	60 - 141
PFTrDA	52 – 145	25	49 – 148	52 - 140
PFTeDA	49 - 158	25	47 – 161	52 - 156
PFBS	66 – 141	20	62 – 144	63 - 145
PFPeS	66 – 144	25	59 – 151	58 - 144
PFHxS	62 – 141	25	57 – 146	44 - 158
PFHpS	59 – 148	25	55 – 152	51 - 150
PFOS	61 – 145	20	58 – 149	43 - 162
PFNS	57 – 143	25	52 – 148	46 - 151
PFDS	56 – 142	25	51 – 147	50 - 144
PFDoS	41 – 140	30	36 – 145	30 - 138
4:2FTS	77 – 135	25	67 – 146	52 - 158
6:2FTS	75 – 137	30	61 – 151	48 - 158
8:2FTS	79 – 136	30	63 – 152	46 - 165
PFOSA	65 – 144	20	61 – 148	47 - 163
NMeFOSA	76 – 132	25	63 – 145	54 - 155
NEtFOSA	75 – 129	25	65 – 139	49 - 156
NMeFOSAA	69 – 134	25	58 – 144	32 - 160
NEtFOSAA	65 – 140	25	59 – 146	51 - 154
NMeFOSE	79 – 129	20	71 – 136	56 - 151
NEtFOSE	79 – 126	25	69 – 137	60 - 147
HFPO-DA	72 – 135	25	63 – 144	58 - 154
ADONA	75 – 138	20	68 – 146	61 - 148
PFMPA	55 – 141	25	51 – 145	48 - 150
PFMBA	59 – 145	20	55 – 148	49 - 154
NFDHA	63 – 146	35	48 – 161	47 - 160
9Cl-PF3ONS	72 – 140	30	56 – 156	44 - 167
11Cl-PF3OUdS	61 – 140	35	46 – 156	36 - 158
PFEESA	57 – 149	20	56 – 151	56 - 144
3:3FTCA	66 – 126	20	62 – 129	32 - 161
5:3FTCA	68 – 130	20	63 – 134	39 - 156
7:3FTCA	55 – 133	25	50 – 138	36 - 149
$^{13}C_4$-PFBA	10 – 130	30	10-130	10-130
$^{13}C_5$-PFPeA	35 – 150	30	40 -150	40 -150
$^{13}C_5$-PFHxA	55 – 150	30	40 -150	40 -150
$^{13}C_4$-PFHpA	55 – 150	30	40 -150	40 -150
$^{13}C_8$-PFOA	60 – 140	30	30-140	30-140
$^{13}C_9$-PFNA	55 – 140	30	30-140	30-140
$^{13}C_6$-PFDA	50 – 140	30	20-140	20-140

Attachment "" to "US Eurofins US Lancaster Laboratories Environmental - Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633 " Page 117 of 121
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ATTACHMENT 21

Table 5. IPR/OPR/LLOPR Acceptance Limits for Wastewater Samples

	Aqueous Matrix [1, 2]			
	IPR			
Compounds	Recovery (%)	RSD (%)	OPR Recovery (%)	LLOPR Recovery (%)
$^{13}C_7$-PFUnA	30 – 140	30	20-140	20-140
$^{13}C_2$-PFDoA	10 – 150	30	10-150	10-150
$^{13}C_2$-PFTeDA	10 – 130	30	10-130	10-130
$^{13}C_3$-PFBS	55 – 150	30	25-150	25-150
$^{13}C_3$-PFHxS	55 – 150	30	25-150	25-150
$^{13}C_8$-PFOS	45 – 140	30	20-140	20-140
$^{13}C_2$-4:2FTS	60 – 200	30	25-200	25-200
$^{13}C_2$-6:2FTS	60 – 200	30	25-200	25-200
$^{13}C_2$-8:2FTS	50 – 200	30	25-200	25-200
$^{13}C_8$-PFOSA	30 – 130	30	10-130	10-130
D_3-NMeFOSA	15 – 130	30	10-130	10-130
D_5-NEtFOSA	10 – 130	30	10-130	10-130
D_3-NMeFOSAA	45 – 200	30	10-200	10-200
D_5-NEtFOSAA	10 – 200	30	10-200	10-200
D_7-NMeFOSE	10 – 150	30	10-150	10-150
D_9-NEtFOSE	10 – 150	30	10-150	10-150
$^{13}C_3$-HFPO-DA	25 – 160	30	25-160	25-160

[1] The recovery limits apply to the target analyte results for IPR, OPR, and LLOPR samples for wastewater matrices. Data for this matrix type are derived from the multi-laboratory validation study and are therefore the limits required for this method.

[2] The recovery limits for the EIS compounds were derived by EPA from the wastewater sample data from multi-laboratory validation study. To simplify laboratory operations, EPA has applied the same EIS recovery limits used for field sample analyses to the EIS recoveries in the IPR, OPR, and LLOPR samples.

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Table 8. QC Acceptance Limits for EIS Recoveries in Wastewater Samples

EIS Compound	Recovery Range (%)
$^{13}C_4$-PFBA	10 – 130 *
$^{13}C_5$-PFPeA	35 - 150
^{13}C5-PFHxA	55 - 150
$^{13}C_4$-PFHpA	55 - 150
$^{13}C_8$-PFOA	60 - 140
$^{13}C_9$-PFNA	55 - 140
$^{13}C_6$-PFDA	50 - 140
$^{13}C_7$-PFUnA	30 - 140
$^{13}C_2$-PFDoA	10 - 150
$^{13}C_2$-PFTeDA	10 – 130 *
$^{13}C_3$-PFBS	55 - 150
$^{13}C_3$-PFHxS	55 - 150
$^{13}C_8$-PFOS	45 - 140
$^{13}C_2$-4:2FTS	60 – 200 *
$^{13}C_2$-6:2FTS	60 - 200 *
$^{13}C_2$-8:2FTS	50 – 200 *
$^{13}C_8$-PFOSA	30 – 130
D_3-NMeFOSA	15 – 130
D_5-NEtFOSA	10 – 130
D_3-NMeFOSAA	45 – 200 *
D_5-NEtFOSAA	10 – 200
D_7-NMeFOSE	10 – 150 *
D_9-NEtFOSE	10 – 150 *
$^{13}C_3$-HFPO-DA	25 - 160

* In the multi-laboratory validation study data for wastewater matrices, some laboratories had difficulties achieving EIS recoveries in this range.

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Table 5. IPR/OPR/LLOPR Acceptance Limits for Target Analytes in Aqueous Matrices

Target Analyte	IPR		OPR/LLOPR Recovery (%)[1]
	Mean Recovery (%)[1]	RSD (%)	
PFBA	70 - 135	21	70 - 140
PFPeA	70 - 135	23	65 - 135
PFHxA	70 - 135	24	70 - 145
PFHpA	70 - 135	28	70 - 150
PFOA	65 - 155	27	70 - 150
PFNA	70 - 140	28	70 - 150
PFDA	65 - 140	26	70 - 140
PFUnA	70 - 135	29	70 - 145
PFDoA	70 - 130	21	70 - 140
PFTrDA	60 - 145	29	65 - 140
PFTeDA	70 - 145	27	60 - 140
PFBS	70 - 140	23	60 - 145
PFPeS	70 - 135	25	65 - 140
PFHxS	70 - 135	27	65 - 145
PFHpS	70 - 140	30	70 - 150
PFOS	70 - 140	29	55 - 150
PFNS	70 - 135	29	65 - 145
PFDS	70 - 135	30	60 - 145
PFDoS	45 - 135	35	50 - 145
4:2FTS	70 - 135	27	70 - 145
6:2FTS	70 - 135	32	65 - 155
8:2FTS	70 - 140	33	60 - 150
PFOSA	70 - 135	22	70 - 145
NMeFOSA	70 - 135	30	60 - 150
NEtFOSA	70 - 130	26	65 - 145
NMeFOSAA	65 - 140	32	50 - 140
NEtFOSAA	70 - 135	28	70 - 145
NMeFOSE	70 - 135	29	70 - 145
NEtFOSE	70 - 130	21	70 - 135
HFPO-DA	70 - 135	23	70 - 140
ADONA	70 - 135	23	65 - 145
PFMPA	60 - 140	23	55 - 140
PFMBA	65 - 145	27	60 - 150
NFDHA	65 - 140	37	50 - 150
9Cl-PF3ONS	70 - 145	30	70 - 155
11Cl-PF3OUdS	50 - 150	35	55 - 160
PFEESA	70 - 135	25	70 - 140
3:3FTCA	70 - 130	23	65 - 130
5:3FTCA	70 - 130	24	70 - 135
7:3FTCA	55 - 130	34	50 - 145

[1] The recovery limits apply to the target analyte results for IPR, OPR, and LLOPR samples for aqueous matrices. Data for this matrix type are derived from the multi-laboratory validation study and are therefore the limits required for this method.

Table 6. Acceptance Limits for EIS and NIS Compounds in All Aqueous Matrices and QC Samples	
EIS Compound	**Recovery (%)[1]**
$^{13}C_4$-PFBA	5[2] - 130
$^{13}C_5$-PFPeA	40 - 130
$^{13}C_5$-PFHxA	40 - 130
$^{13}C_4$-PFHpA	40 - 130
$^{13}C_8$-PFOA	40 - 130
$^{13}C_9$-PFNA	40 - 130
$^{13}C_6$-PFDA	40 - 130
$^{13}C_7$-PFUnA	30 - 130
$^{13}C_2$-PFDoA	10 - 130
$^{13}C_2$-PFTeDA	10 - 130
$^{13}C_3$-PFBS	40 - 135
$^{13}C_3$-PFHxS	40 - 130
$^{13}C_8$-PFOS	40 - 130
$^{13}C_2$-4:2FTS	40 - 200
$^{13}C_2$-6:2FTS	40 - 200
$^{13}C_2$-8:2FTS	40 - 300
$^{13}C_8$-PFOSA	40 - 130
D_3-NMeFOSA	10 - 130
D_5-NEtFOSA	10 - 130
D_3-NMeFOSAA	40 - 170
D_5-NEtFOSAA	25 - 135
D_7-NMeFOSE	10 - 130
D_9-NEtFOSE	10 - 130
$^{13}C_3$-HFPO-DA	40 - 130
$^{13}C_3$-PFBA	50 - 200
$^{13}C_2$-PFHxA	
$^{13}C_4$-PFOA	
$^{13}C_5$-PFNA	
$^{13}C_2$-PFDA	
$^{18}O_2$-PFHxS	
$^{13}C_4$-PFOS	

[1] The recovery limits for the EIS compounds were derived by EPA from the aqueous sample data from multi-laboratory validation study. To simplify laboratory operations, EPA has applied the same EIS recovery limits used for field sample analyses to the EIS recoveries in the IPR, OPR, and LLOPR samples. There are no IPR mean or RSD criteria for the EIS compounds.

[2] Recovery of $^{13}C_4$-PFBA can be problematic in some field samples. Although the lower limit for recovery for this EIS is set below 10%, laboratories should routinely track recovery of this EIS and take reasonable steps to ensure that recovery is at least 10% in the majority of samples.

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eurofins	Always check on-line for validity. **Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633**	Level: **Work Instruction**
Document number: **T-PFAS-WI49211**		
Old Reference:		
Version: **5**		Organisation level: **5-Sub-BU**
Approved by: **ULJC** Effective Date: **21-DEC-2023**	Document users: **5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep**	Responsible: **5_EUUSLA_PFAS_Manager**

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Revision Log

	Revision: 05	**Effective date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout document	Enhancement	Added references to Draft 4. Removed references to Sciex 4500.
Apparatus and Equipment	Enhancement	Added "or equivalent" and missing part numbers; updated WAX cartridge
Reagents and Standards	Enhancement	Added "or equivalent" and missing part numbers. Corrected the percent purity.

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	Revision: 05	**Effective date:** This version
Section	**Justification**	**Changes**
Calibration A	Method Requirement	Added S/N for compounds without confirmation ions Removed verbiage regarding Linear/Branched standard
Calibration A	Clarification	Removed WV requirements
Procedure B/C	Clarification	Corrected the Envi-carb and filtration steps done in in the instrument room. Corrected IS name.
Procedure D	Method Requirement	Instrument Blanks report to MDL
Procedure D	Enhancement	Added reporting procedure for samples with failing EIS and NIS
Procedure D	Clarification	Updated NIS criteria, updated RT criteria
Procedure D	Clarification	Mass tune must be +/- 0.2 amu
Attachment 3	Enhancement	Updated to reflect current method
Attachment 5-20	Enhancement	updated attachments to reflect new standards

Revision:	4	**Effective date:** 09.MAY.2023
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Enhancement	updated to version 3 of draft
Procedure	Reflects current procedure	clarify procedure when IDAs fail, add DUP requirements
Apparatus and Equipment	Enhancement	Proprietary Content
Statistical infromation/Method Performance	Enhancement	Add DUP requirements

Reference

1. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, 4th DRAFT, July 2023.

2. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, 3rd DRAFT, December 2022.

3. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, 2nd DRAFT, June 2022.

4. USEPA Draft Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, DRAFT, August 2021.

5. Chemical Hygiene Plan, current version.

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Cross Reference

Document	Document Title
T-PFAS-WI21568	Manifold and N-EVAP Cleaning for PFAS Extractions
T-PEST-WI9847	Common Equations Used During Chromatographic Analyses
QA-SOP11178	Demonstrations of Capability
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
G-DC-FRM23907	Redacted SOPs

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in solid. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS. Compounds other than those listed may be analyzed by client request.

Analyte	Acronym	CAS#
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnDA	2058-94-8
Perfluoro-n-butanoic acid	PFBA	375-22-4
Perfluoro-n-pentanoic acid	PFPeA	2706-90-3
8:2 - Fluorotelomersulfonic acid	8:2FTS	39108-34-4
N-methylperfluoro-1-octanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethylperfluoro-1-octanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
4:2-Fluorotelomersulfonic acid	4:2-FTS	757124-72-4
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
6:2-Fluorotelomersulfonic acid	6:2-FTS	27619-97-2

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Analyte	Acronym	CAS#
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoDS	79780-39-5
Perfluorooctanesulfonamide	PFOSA	754-91-6
2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	NMePFOSAE	24448-09-7
N-methylperfluoro-1-octanesulfonamide	NMePFOSA	31506-32-8
2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	NEtPFOSAE	1691-99-2
N-ethylperfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid; (Hexafluoropropylene oxide dimer acid)	HFPODA	13252-13-6
Ammonium 4,8-dioxa-3H-perfluorononanoic acid	DONA **	919005-14-4 *
Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS, F53B major	756426-58-1 *
Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS, F53B minor	763051-92-9 *
3-Perfluoropropylpropanoic acid	3:3 FTCA	356-02-5
3-Perfluoropentylpropanoic acid	5:3 FTCA	914637-49-3
3-Perfluoroheptylpropanoic acid	7:3 FTCA	812-70-4
Perfluoro-3-methoxypropanoic acid	PFMPA, PFECA F	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA, PFECA A	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	PFECA B	151772-58-6
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA, PES	113507-82-7

*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

Basic Principles

A solid sample is fortified with isotopically-labeled extraction standards. The sample extract is shaken, centrifuged, and the supernatant decanted. Carbon cleanup is performed on each sample extract. Sample extract is diluted to volume and then concentrated. The sample is then passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. The extract is fortified with Isotopically-labeled injection internal standards and filtered. It is then analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Interferences

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Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where possible.

2. A precolumn, Phenomenex Luna, 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.

3. All parts of the SPE manifold must be cleaned as per *T-PFAS-WI21568*.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

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The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. IDOC trials are spiked at the OPR Level.

See *QA-SOP11178* for additional information on IDOC and DOC.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 4-oz PP specimen containers with polyethylene screw caps. Keep the sample sealed from time of collection until extraction.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

B. Sample Storage and Shipment

1. Solid and Biosolid samples must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when the samples are received at the laboratory.

2. Solid and Biosolid samples stored in the lab must be protected from light and held at a temperature of 0° to 6°C, or less than or equal to -20°C, until extraction.

3. Solid and Biosolid samples must be extracted within 90 days. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C and protected from light.

NOTE: Biosolid samples stored under refrigeration may produce gases that may cause sample to be expelled from the container when opened. This may produce noxious odors. It is recommended to store frozen if extraction will not occur for a few days.

Apparatus and Equipment

A. Apparatus

1. Specimen container; PP; natural; graduated; non-sterile; separate 1/4-turn red screwcap; 4 oz; 600 EA, Greenwood Products, Inc. Part No. HP30011 or equivalent

2. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Medline Catalog No. MLAB15021 or equivalent

3. 10-mL polypropylene volumetric flask, Class A – Fisher Scientific, Catalog No. S02288 or equivalent.

4. HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Catalog No. 1000-1902-PC.

5. Analytical Balance – Capable of weighing to 0.0001 g

6. Top-Loading Balance – Capable of weighing to 0.01 g

7. Solid phase extraction (SPE) Cartridge; Strata-X; X-AW, Weak Anion Mixed Mode; 500mg/6ml; sorbent; 33um; 30 EA. Catalog No. 8B-S038-HCH or equivalent.

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8. Large-volume SPE Reservoir (25-mL) - Millipore-Sigma; Product No. 54258-U or equivalent.
9. SPE Tube Adapter - Millipore-Sigma; Product No. 57020-U or equivalent.
10. SPE vacuum extraction manifold –"Resprep" 24-port manifold; Restek Corp Catalog No. 26080, or equivalent.
11. Polypropylene SPE delivery needles – Agilent; Catalog No. 12234511 or equivalent.
12. Centrifuge – "Q-Sep 3000"; Restek Corp. Catalog No. 26230, or equivalent, capable of a minimum rotational speed of 3000 rpm.
13. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000-µL. Fisher Scientific Catalog No. 14-287-150, or equivalent.
14. Polypropylene pipette tips: 0-200µl. Fisher; Catalog No. 02-681-135 or equivalent
15. Polypropylene pipette tips: 101-1000µl. Fisher, Catalog No. 02-707-507 or equivalent
16. Pipettes – Disposable transfer. FisherScientific, Catalog No. 13-711-7M or equivalent
17. Vortex mixer, variable speed, Fisher Scientific or equivalent.
18. N-Evap sample extract concentrator with N_2 supply and water bath for temperature control. Organomation, Inc. Catalog No. 11250, or equivalent.
19. Reagent Water Purification System: Capable of producing ultrapure "Type 1/Milli-Q"-grade water from in-house deionized water system. Millipore SAS; Catalog No. FTPF08831.
20. Thermo Target PP Polyspring inserts, Catalog No. GEME03CPPSP or equivalent
21. Short-Cap Vial with Grad Marking Spot, 9-425 Screw-Thread, 2.0 mL, 9 mm, Clear, 1000-pk., Restek Catalog No. 21141 or equivalent.
22. Short Screw Caps, Polypropylene, Screw-Thread, PTFE/Silicone Septa, Blue, Preassembled, 2.0 mL, 9 mm, 100-pk, Restek Catalog No. 24485 or equivalent.
23. Centrifuge tubes – 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Catalog No. 06-443-21 or equivalent
24. Polypropylene bottles for standard storage - 4 mL; Fisher Scientific, Catalog No. 2006-9125
25. Stainless steel spatula/scoop set. Bel-Art SP Scienceware; Product No. 11-865-130.
26. pH paper - Hydrion, range 5.5-8.0. Fisher Scientific, Catalog No. 14-853-275 or equivalent.
27. Syringe filter - Restek, 25 mm Syringe Filter, 0.22 um, 100-pk. Catalog No. 26148 or equivalent.
28. Silanized glass wool - Sigma-Alrich, Catalog No. 20411 or equivalent
29. Glass fiber filter, 47 mm, 1 um, PALL A/E or equivalent
30. Variable speed mixing table (Fisherbrand Nutating mixer or equivalent)
31. Wooden Tongue Depressors - Fisher; Catalog No. 11-700-555, or equivalent.

B. Equipment

1. AB Sciex Triple Quad 5500/5500 Plus Turbo V Ion Source or equivalent

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ExionLC Controller
ExionLC AC Pump
ExionLC AC Autosampler
Exion AC Column Oven
Data system – Analyst 1.6.3

2. Proprietary Content

3. HPLC columns

   Analytical column: Gemini 3µm C18, 50 x 3 mm, Phenomenex Catalog No. 00B-4439-Y0 or equivalent
   Pre-column: Luna, 5µm C18(2), 50 x 3 mm, Phenomenex Catalog No. 00B-4252-Y0, or equivalent

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

A. Reagents:

1. Methanol (MeOH) – VWR "Chromasolv LC-MS" grade Catalog No. BJ34966-4L or equivalent

2. Acetonitrile (ACN) – Fisher Scientific, Optima Catalog No. A955-4 or equivalent

3. Ammonium acetate – Sigma Aldrich, Catalog No. 431311-250G or equivalent. Stored at 2-8°C.

4. Ammonium hydroxide, 30% in water, certified ACS+ grade or equivalent, store at room temperature. VWR Catalog No. EMD-AX1303-3 or equivalent

5. Methanolic ammonium hydroxide (0.3%) – add ammonium hydroxide (10 mL, 30%) to methanol (990 mL), store at room temperature, replace after 1 month

6. Methanolic ammonium hydroxide (1%) - add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month

7. Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid - add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month.

8. Acetic Acid – ACS grade or equivalent, store at room temperature, Sigma Aldrich 338826-100ML or equivalent.

9. Acetic Acid (0.1%) – dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.

10. Formic acid - Honeywell, Catalog No. 56302-50ML-GL or equivalent

    a. Formic acid (aqueous, 0.1 M) - dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years

    b. Formic acid (aqueous, 0.3 M) - dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years

    c. Formic acid (aqueous, 5% v/v) - mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years

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d. Formic acid (aqueous, 50% v/v) - mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years

e. Formic acid (methanolic 1:1, 0.1 M formic acid/methanol) - mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years

11. "Superclean Envi-Carb"; bulk sorbent. Millipore-Sigma; 50g; Product # 57210-U.

12. Solids reference matrix – Ottawa or reagent-grade sand

13. 20mM ammonium acetate solution in 95:5(v/v) Milli-Q water/acetonitrile-Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 1900 mL Milli-Q water and mix well to dissolve the ammonium acetate.Add 100 mL acetonitrile and mix well.Store at room temperature for up to 2 months.Different volumes can be prepared as long as final concentrations are equivalent.

14. 20 mM ammonium acetate solution in 90:10 acetonitrile/Milli-Q water – Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 200 mL of Milli-Q water and mix well to dissolve the Ammonium Acetate. Add 1800 mL of acetonitrile and mix well. Store at room temperature for up to 2 months.Different volumes can be prepared as long as final concentrations are equivalent.

B. Standards:

Standards are prepared using calibrated pipettes, polypropylene microcentrifuge tubes, polypropylene bottles, and 10-mL Class A PP volumetric flasks to create solutions at desired concentrations. The concentrated solution is injected below the surface of the diluting solvent. After preparation is completed, standards should be vortexed to ensure complete mixing. Measurement of volumes less than 5 µl should be avoided in routine production operations.

All stock, intermediate and spiking solutions are prepared using Methanol.

All initial calibration, initial calibration verification, and linear branched working standard solutions are prepared using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid.

All diluted solutions must be stored in HDPE containers that have been thoroughly rinsed with methanol.

Stock standard and intermediate standard solutions are stored in the refrigerator in labeled polypropylene screw-top vials, PP bottles, or PP centrifuge tubes.

Expiration dates are managed through LIMS Reagent Module. Solutions transferred from sealed glass ampules to screw-capped vials are given expiration dates of 1 year from the date opened or the expiration date provided by the vendor, whichever occurs sooner. Intermediate solutions are given an expiration date of 6 months from the preparation date, or the expiration date from the ampule provided by the vendor, whichever occurs sooner. The ampules and transferred solutions are stored in the refrigerator.

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator. When these solutions are prepared they must be tested prior to use in the PFAS extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

Working initial calibration solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working initial calibration solution, whichever occurs sooner.

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The primary/preferred standard vendor is Wellington Laboratories, Inc. Ontario, Canada. Listed catalog numbers are taken from Wellington product lists. Equivalent standards may be substituted, if the listed standards are unavailable.

The solution concentration listed is as presented on the certificate of analysis and includes adjustment for purity and the salt form of the compound used.

Note: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS, and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See *Attachment 4*.
If the compound purity is assayed to be 98% or greater, weight can be used without correction to calculate concentrations.

Log purchased standards into LIMS Reagent. Select the solution category SOURCE for purchased mixes and/or single-compound ampules. LIMS Reagent system will assign formatted names to the purchased standard solutions. The automatically-generated name can be overwritten with a manually created name if desired. Use labels printed through the LIMS Reagent to identify and track standard solutions after transfer from original ampule to storage vial. The C of A for the ampulated stock standard is attached in LIMS Reagent for reference.

Standards are prepared by transferring a known quantity of Standard to a final volume of solvent. Standard Preparation is documented in LIMS Reagent. Solutions are stored by Type in LIMS Reagent, i.e.,INTERMEDIATE=working solutions and intermediate standards and SOURCE=stocks (ampulated solutions). Each Standard is given a unique name.

The following attachments provide examples of standard preparation and purchasing information. Refer to the documentation in LIMS Reagent for standards preparation information.
Attachment 5 - 1633 Native PFAS Intermediate A
Attachment 6 - 1633 Native PFAS Intermediate B
Attachment 7 - 1633 Extracted Internal Standard Intermediate
Attachment 8 - 1633 Non-Extracted Internal Standard Intermediate
Attachment 9 - 1633 TOP Surrogate Intermediate
Attachment 10 - 1633 ICAL Standard Preparation Scheme
Attachment 11 - 1633 Initial Calibration Standard Concentrations
Attachment 12 - Bile Salts Stock Solution
Attachment 13 - Bile Salts Intermediate
Attachment 14 - Bile Salts Working Solution
Attachment 15 - 1633 WDM Standard
Attachment 16 - 1633 Native Mid-Level Spike
Attachment 17 - 1633 Native Low-Level Spike Intermediate
Attachment 18 - 1633 Native Low-Level Spike
Attachment 19 - 1633 ICV Working Standard
Attachment 20 - 1633 Purchased/Ampulated Standards

Calibration

A. Initial Calibration

1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order curve fit (quadratic). In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, NEtFOSAA, PFNA, PFOA, NEtFOSE, NMeFOSE, NEtFOSA, and NMeFOSA. S/N ratio for the lowest standard within the quantitation range must be greater than or equal to 3:1 for quantification ions and the confirmation ions, or greater than or equal to 10:1 if the analyte only has a quantification ion.

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2. Analyze a Cal4 level standard that contains Bile Salts retention time marker. The analysis of this standard is used to evaluate the interference from bile salts in tissue samples. This will assist the chemist in identifying and properly integrating this compound in samples.

Example Initial Calibration Sequence:

1. Instrument Blank
2. Instrument Blank
3. Instrument Blank
4. CAL 1
5. CAL 2
6. CAL 3
7. CAL 4
8. CAL 5
9. CAL 6
10. CAL 7
11. ICB (Instrument Blank)
12. ICV
13. WDM (Bile Salts standard)

3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See *Attachment 2* for additional information about compound relationships.

4. Analyze a standard containing 100 ppb of Taurodeoxycholic Acid (TDCA), Taurochenodeoxycholic Acid (TCDCA), and Tauroursodeoxycholic Acid (TUDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in samples. The analytical conditions must be set to allow a separation of at least 1 minute between TDCA and PFOS.

Note: For better accuracy, PFTrDA is quantitated using the average of the areas of labeled compounds 13C2-PFTeDA and 13C2-PFDoDA.

Compound	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA

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Compound	Extraction Standard
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

5. Fit the curve.

   a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.

   b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for the compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%.

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c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within ±30% of its true value.

d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS, and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See *Attachment 4*.

6. Initial Calibration Verification (ICV)

A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock by a second analyst may be used. The calculated amount for each analyte must be within ±30% of the true value. If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.

2. Acceptance criteria

a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

A. Sample Preparation

NOTE: Prior to weighing out samples, thoroughly mix each sample using a wooden tongue depressor or stainless steel spoon to ensure a homogeneous sample matrix. Stir from the bottom to the top in a circular motion along the sides of the jar, breaking particles to less than 1 mm by pressing against the side of the container. Remove rocks, invertebrates, and foreign objects. Vegetation can either be removed or cut into smaller pieces based on project requirements.

1. On a calibrated, top-loading balance, accurately weigh:

- 5.0g ± 0.10g (dry weight; note: maximum 10g sample aliquot)
- 0.5g for biosolids (dry weight; note: maximum 1g sample aliquot)

of solid sample into a tared, labeled 50-mL centrifuge tube using a disposable polypropylene spatula. Record sample weight in the prep entry system.

2. For each batch - maximum 20 samples - include the following quality control samples:

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a. Method Blank: Weigh 5.0g ± 0.10g (0.5g for biosolids) of sand wetted with 2.5g (0.25g for biosolids) of reagent water

b. LCS: Fortify 5.0g ± 0.10g (0.5g for biosolids) of sand wetted with 2.5g (0.25g for biosolids) reagent water and spiked with 200 µL of Native 1633 Mid-Level Spike Solution (PFC_1633_MID_XXXXX).

c. LLCS: Fortify 5.0g ± 0.10g (0.5g for biosolids) of sand wetted with 2.5g (0.25g for biosolids) reagent water and spiked with 400 µL of Native 1633 Low-Level Spike Solution (PFC_1633_LOW_XXXXX).

d. Matrix Spike/Matrix Spike Duplicate (MS/MSD): Fortify 5.0g ± 0.10g (dry weight; note: maximum 10g sample aliquot) (0.5g for biosolids (dry weight; note: maximum 1g sample aliquot)) of sample as specified in sample preparation log with 200 µL of Native 1633 Mid-Level Spike Solution (PFC_1633_MID_XXXXX).

e. Matrix Duplicate (MD): Weigh 5.0g ± 0.10g (dry weight; note: maximum 10g sample aliquot) (0.5g for biosolids (dry weight; note: maximum 1g sample aliquot)) of sample as specified in sample preparation log.

3. Add 25 µl working labeled extraction standard spike solution (PFC_1633_SS_XXXXX) to each sample/QC tube.

4. Cap and vortex for approximately 30 seconds.

5. Allow samples/QC to equilibrate for at least 30 minutes.

6. Add 10 mL of 0.3% methanolic ammonium hydroxide to each centrifuge tube.

7. Cap and vortex.

8. Shake for 30 minutes on a variable speed mixing table.

9. Centrifuge for 10 minutes and transfer supernatant to a clean 50 mL polypropylene centrifuge tube.

10. Add 15 mL of 0.3% methanolic ammonium hydroxide to the remaining solid sample in each centrifuge tube. Cap and vortex.

11. Shake for 30 minutes on a variable speed mixing table.

12. Centrifuge for 10 minutes and decant the supernatant from the second extraction into the centrifuge tube with the supernatant from the first extraction.

13. Add another 5 mL of 0.3% methanolic ammonium hydroxide to the remaining sample in each centrifuge tube.

14. Vortex to mix.

15. Centrifuge for 10 minutes and decant the supernatant from the third extraction into the centrifuge tube with the supernatant from the first and second extraction.

16. Using a 10mg scoop, add 10mg of Superclean Envi-Carb to the combined extract. Vortex for 30 seconds.

17. Centrifuge for 10 minutes.

18. Decant the extract from each tube into a clean, labeled, 250-mL HDPE bottle making sure to leave the Superclean Envi-Carb behind. Bring the 250-mL HDPE container up to 250 mL with Milli-Q water.

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19. Check that the pH is 6.5 ± 0.5 and adjust as necessary with 50% formic acid or 30% ammonium hydroxide (or with 5% formic acid and 3% aqueous ammonium hydroxide). **NOTE: Never put pH test strips into the sample. Use a disposable pipette to place a few drops of sample onto the pH paper.**

20. Invert container to thoroughly mix.

B. Solid Phase Extraction (SPE)- Manual Sample Extraction

1. Pack clean silanized glass wool to half the height of the WAX SPE cartridge barrel.

2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.

3. Condition each SPE cartridge with the following reagents in the following order:

   a. 15 mL 1% methanolic ammonium hydroxide

   b. 5 mL 0.3M formic acid

   c. Discard conditioning eluent(s)

4. Label each sample bottle, cap and reservoir with the same number to insure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).

5. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges. Allow full volume to pass through each cartridge by gravity, if possible. Apply light vacuum if necessary. The drip rate should be approximately 1-2 drops per second.

6. Rinse the walls of the bottle with 5mL reagent water (twice) followed by 5 mL of **1:1 0.1M formic acid/methanol** and pass the rinses through the cartridge using vacuum. Dry the cartridge by pulling air through for 15 seconds. Discard the rinse solution.

7. After full volume has passed through the cartridges, dry the cartridges with vacuum - no more than 15" of Hg - for approximately five minutes. After five minutes, visually inspect the cartridge to determine if the sorbent is dry. This done by comparing the cartridge to a visual standard (an unused SPE cartridge). If the sorbent is not dry, continue to check at one minute intervals until the cartridge is dry.

8. Discard the waste and rinse the waste reservoir with DI water. Wipe each needle with a Kim-wipe/methanol.

9. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge.

10. Add 5 mL of 1% methanolic ammonium hydroxide to the sample bottle.

11. Transfer the rinse to the SPE reservoirs, washing the walls of the reservoirs.

12. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.

13. Disconnect the cartridge/adapter from the manifold. Remove the collection tubes.

14. Add 25 µL of concentrated acetic acid to each collection tube and vortex to mix thoroughly.

15. Place extracts in the cooler.

Note: The instrument lab chemist performs the next steps.

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16. Bring each sample extract to final volume 5mL with methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.

17. Add 25 µL of Internal Standard Spike Solution (PFC_1633_IS_XXXXX) to a clean 15-mL polypropylene centrifuge collection tube.

18. Cap and vortex to mix.

19. Place a syringe filter (25-mm filter, 0.2-µm nylon membrane) on a 3-mL polypropylene syringe. Take the plunger out and carefully decant ~1 mL the sample supernatant into the syringe barrel. Replace the plunger and filter ~1 mL of sample into the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.

20. Cap the centrifuge tube. Store the remaining centrifuged extracts in the refrigerator for dilution or reinjection if needed.

C. Proprietary Content

1. Proprietary Content
2. Proprietary Content
3. Proprietary Content
4. Proprietary Content
5. Proprietary Content
6. Proprietary Content
7. Proprietary Content
8. Proprietary Content
9. Proprietary Content
10. Proprietary Content
11. Proprietary Content
12. Proprietary Content
13. Proprietary Content
14. Proprietary Content
15. Proprietary Content
16. Proprietary Content

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D. LC/MS/MS Analysis

1. Mass Calibration and Tuning

    a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

    b. When masses fall outside of the ±0.2 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.2 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.

2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (5500/5500 Plus7500) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

3. Example acquisition method: See *Attachment 3*. Mass Transitions: See *Attachment 1*.

4. Instrument Sensitivity Check (ISC) and Instrument Blanks

    a. Prior to sample analysis, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within ±30% of their true value. The signal-to-noise ratio must be greater than or equal to 3:1 for the quantitation ions and confirmation ions, and 10:1 for quantitation ions that have no confirmation ion. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.

    b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than the MDL. If acceptance criteria are not met, the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.

5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.

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6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, bile salt marker, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes Bile Salts RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

   Example Sample Sequence:

   1. Instrument blank
   2. Instrument blank
   3. Instrument blank
   4. Instrument Sensitivity Check (CCV0_CAL1)
   5. CCV 2_CAL4
   6. Bile Salt marker (WDM)
   7. Instrument Blank (ICB)
   8. Method Blank (MB)
   9. Low Level LCS (LLCS)
   10. LCS
   11. Sample (10 or less)
   12. CCV 3_CAL4
   13. Instrument Blank

7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extracted method blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted. If there is no target analyte greater than the reporting limit in samples associated with an unacceptable method blank, the data may be reported with qualifiers.

9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.

   a. LLCS, LCS, MS, extraction standard recoveries, and RPDs are calculated and compared to the limits stored on the LIMS.

   b. If LLCS and LCS recoveries are acceptable, proceed to sample quantitation.

   c. If the LCS and LLCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, consult supervisor for course of action based on batch and sample results.

   d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

10. Calculate the relative percent difference (RPD) between the sample and the matrix duplicate (DUP). The RPD must be less than or equal to 30%. This criteria only applies to analytes whose concentration in the sample is greater than or equal to the LOQ. If the criteria is not met, narrate with an NCM and report the data.

11. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, MS/MSD, and sample duplicate prior to extraction. The recovery of the extraction standards should

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be within QC acceptance criteria. (See Statistical Information/Method Performance section of this SOP.) Quantitation by isotope dilution generally precludes any adverse effect on data quality due to IDA recoveries being outside of the acceptance limits as long as the signal-to-noise ratio is greater than 10:1.

12. If the extraction standard recovery(ies) is(are) outside the QC limit(s), dilute the sample extract and re-analyze. If the EIS recovery(ies) of the diluted sample extract are below 5%, a reduced sample aliquot will be extracted. If the EIS recovery(ies) of the diluted sample extract are greater than 5%, the data is reported.

13. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 50-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 50-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.

14. Compare the retention times of all of the analytes, surrogates, and internal standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than 0.4 minutes. Furthermore, native compounds with an exact isotopically-labeled analogue must elute within 0.1 minutes from their associated analogue.

    If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.

15. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of PFBA, PFECA F(PFMPA), PFECA A(PFMBA), PFPeA, NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required.

16. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of Internal Standard Spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%.Extracts requiring greater than a 10x dilution should be reextracted using a reduced aliquot.

    Dilution Example 1/10: Mix 895 µL of Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution with 100 µl of sample extract and 5 µL of Internal Standard Spike solution. Vortex to mix. Using an auto-pipette, transfer an aliquot of the mixed solution into a labeled auto-sampler vial. Cap and vortex thoroughly to mix.

Calculations

1. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

$$\text{On-column Concentration} = \text{peak area ratio} \div \text{AVE RRF}$$

3. On-Column Analyte Concentration using linear curve

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On-column Concentration = (peak area ratio - intercept) ÷ slope

4. Sample Concentration

   Sample concentration (ng/g) = (On-column concentration x Final Sample Volume x DF) ÷ Initial Sample Volume

5. Ion Ratio

   ion ration = (peak area or height of quantifier)/(peak area or height of qualifier)

6. See *T-PEST-WI9847* for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

The LCS should contain all compounds of interest. LCS, MS, and extraction standard recoveries are compared to the limits stored on the LIMS. These limits are method defined.

QC parameter	Lower acceptance limit	High acceptance limit
Extracted Internal standard (EIS)	20%	150%
Non-extracted Internal Standard (NIS)	50% of the average NIS from the initial calibration	200%
Analyte recoveries LCS/LLCS/MS/MSD	40%	150%
Sample/Duplicate	</= 30%	NA

Note: lower acceptance limit for EIS cannot not be <20%, lower acceptance limit for analyte recovery cannot be <40%.

Historical data for MS/Ds, LCSs, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to *QA-SOP11892* for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank (sand), an LCS/LLCS (sand spiked with all compounds to be determined carried through the entire procedure), and a sample/Dup, must be extracted and analyzed. MS/MSD is extracted only if submitted by the client. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

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Attachment:
Attachment 01 - Mass Transitions (.doc)
Attachment 02 - Standards relationships (.docx)
Attachment 03 - Acquisition Method (.pdf)
Attachment 04 - Example Certificate of Analysis (.pdf)
Attachment 05 – 1633 Native PFAS Intermediate A (.pdf)
Attachment 06 – 1633 Native PFAS Intermediate B (.pdf)
Attachment 07 – 1633 EIS Intermediate (.pdf)
Attachment 08 - 1633 Non-Extracted Internal Standard Intermediate (.pdf)
Attachment 09 - 1633 TOP Surrogate Intermediate (.pdf)
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Attachment 18 - 1633 Native Low-Level Spike (.pdf)
Attachment 19 - 1633 ICV Standard (.pdf)
Attachment 20 - 1633 Purchased Standards (.pdf)

G-DC-FRM23907 Redacted SOPs
QA-SOP11178 Demonstrations of Capability
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
T-PEST-WI9847 Common Equations Used During Chromatographic Analyses
T-PFAS-WI21568 Manifold and N-EVAP Cleaning for PFAS Extractions
Attachment: Attachment 01 - Mass Transitions (doc)
Attachment: Attachment 02 - Standards relationships (docx)
Attachment: Attachment 03 - Acquisition Method (pdf)
Attachment: Attachment 04 - Example Certificate of Analysis (pdf)
Attachment: Attachment 05 – 1633 Native PFAS Intermediate A (pdf)
Attachment: Attachment 06 – 1633 Native PFAS Intermediate B (pdf)
Attachment: Attachment 07 – 1633 EIS Intermediate (pdf)
Attachment: Attachment 08 - 1633 Non-Extracted Internal Standard Intermediate (pdf)
Attachment: Attachment 09 - 1633 TOP Surrogate Intermediate (pdf)
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Attachment: Attachment 12 - Bile Salts Stock Solution (pdf)
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Attachment: Attachment 15 - 1633 WDM Standard (pdf)
Attachment: Attachment 16 - 1633 Native Mid-Level Spike (pdf)
Attachment: Attachment 17 - 1633 Native Low-Level Spike Intermediate (pdf)
Attachment: Attachment 18 - 1633 Native Low-Level Spike (pdf)
Attachment: Attachment 19 - 1633 ICV Standard (pdf)
Attachment: Attachment 20 - 1633 Purchased Standards (pdf)

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Version history

Version	Approval	Revision information	
3	01.FEB.2023		
4	09.MAY.2023		
5	07.DEC.2023		

Attachment 1

Mass Transitions AB Sciex 4500/5500/5500+

Compound	Parent Ion	Daughter Ion
13C3-PFBA	216.0	172.0
13C4-PFBA	216.8	171.9
PFBA	212.8	168.9
13C5-PFPeA	268.3	223
PFPeA	263.0	219.0
PFPeA (2)	263.0	68.9
13C3-PFBS	302.1	79.9
13C3-PFBS (2)	302.1	98.9
PFBS	298.7	79.9
PFBS (2)	298.7	98.8
13C2-4:2-FTS	329.1	80.9
13C2-4:2-FTS (2)	329.1	309.0
4:2-FTS	327.1	307.0
4:2-FTS (2)	327.1	80.9
13C2-PFHxA	315.1	270.0
13C2-PFHxA (2)	315.1	119.4
13C5-PFHxA	318.0	273.0
13C5-PFHxA (2)	318.0	120.3
PFHxA	313.0	269.0
PFHxA (2)	313.0	118.9
PFPeS	349.1	79.9
PFPeS (2)	349.1	98.9
18O2-PFHxS	403.0	83.9
13C3-PFHxS	402.1	79.9
13C3-PFHxS (2)	402.1	98.9
PFHxS	398.7	79.9
PFHxS (2)	398.7	98.9
13C4-PFHpA	367.1	322.0
PFHpA	363.1	319.0
PFHpA (2)	363.1	169.0
13C2-6:2-FTS	429.1	80.9
13C2-6:2-FTS (2)	429.1	409.0
6:2-FTS	427.1	407.0
6:2-FTS (2)	427.1	80.9
PFHpS	449.0	79.9
PFHpS (2)	449.0	98.8
13C4-PFOA	417.1	172.0

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Attachment 1

Compound	Parent Ion	Daughter Ion
13C8-PFOA	421.1	376.0
PFOA	413.0	369.0
PFOA (2)	413.0	169.0
13C4-PFOS	502.8	79.9
13C4-PFOS (2)	502.8	98.9
13C8-PFOS	507.1	79.9
13C8-PFOS (2)	507.1	98.9
PFOS	498.9	79.9
PFOS (2)	498.9	98.8
13C5-PFNA	468.0	423.0
13C9-PFNA	472.1	427.0
PFNA	463.0	419.0
PFNA (2)	463.0	219.0
13C8-PFOSA	506.1	77.8
PFOSA	498.1	77.9
PFOSA (2)	498.1	478.0
PFNS	548.8	79.9
PFNS (2)	548.8	98.8
13C2-PFDA	515.1	470.1
13C6-PFDA	519.1	474.1
PFDA	512.9	469.0
PFDA (2)	512.9	219.0
13C2-8:2-FTS	529.1	80.9
13C2-8:2-FTS (2)	529.1	509.0
8:2-FTS	527.1	507.0
8:2-FTS (2)	527.1	80.8
d7-NMePFOSAE	623.2	58.9
NMePFOSAE	616.1	58.9
d3-NMePFOSA	515.0	219.0
NMEPFOSA	511.9	219.0
NMEPFOSA (2)	511.9	169.0
d3-NMeFOSAA	573.2	419.0
NMeFOSAA	570.1	419.0
NMeFOSAA (2)	570.1	483.0
d9-NEtPFOSAE	639.2	58.9
NEtPFOSAE	630.0	58.9
d5-NETPFOSA	531.1	219.0
NEtPFOSA	526.0	219.0
NEtPFOSA (2)	526.0	169.0
PFDS	599.0	79.9

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Attachment 1

Compound	Parent Ion	Daughter Ion
PFDS (2)	599.0	98.8
13C7-PFUnDA	570.0	525.1
PFUnDA	563.1	519.0
PFUnDA (2)	563.1	269.1
d5-NEtFOSAA	589.2	419.0
NEtFOSAA	584.2	419.1
NEtFOSAA (2)	584.2	526.0
13C2-PFDoDA	615.1	570.0
PFDoDA	613.1	569.0
PFDoDA (2)	613.1	319.0
PFDoS	699.1	79.9
PFDoS (2)	699.1	98.8
PFTrDA	663.0	619.0
PFTrDA (2)	663.0	168.9
13C2-PFTeDA	715.2	670.0
PFTeDA	713.1	669.0
PFTeDA (2)	713.1	168.9
13C3-HFPODA	286.9	168.9
13C3-HFPODA (2)	286.9	184.9
HFPODA	284.9	168.9
HFPODA (2)	284.9	184.9
DONA	376.9	250.9
DONA (2)	376.9	84.8
9Cl-PF3ONS	530.8	351.0
9Cl-PF3ONS (2)	532.8	353.0
11Cl-PF3OUdS	630.9	450.9
11Cl-PF3OUdS (2)	632.9	452.9
PFECA B (NFDHA)	295.0	201.0
PFECA B(NFDHA) (2)	295.0	84.9
PFECA F (PFMPA)	229.0	84.9
3:3 FTCA	241.0	177.0
3:3 FTCA (2)	241.0	117.0
PFECA A (PFMBA)	279.0	85.1
PFEESA (PES)	314.8	134.9
PFEESA (PES) (2)	314.8	82.9
5:3 FTCA	341.0	237.1
5:3 FTCA (2)	341.0	217.0
7:3 FTCA	441.0	316.9
7:3 FTCA (2)	441.0	336.9

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Attachment 2

PFAS Injection Standards/Extraction Standards/Native Compounds

Injection Standards

Inj Std	Internal Standard/Injection Standard
I13C3-PFBA	13C3-PFBA
I13C2-PFHxA	13C2-PFHxA
I13C4-PFOA	13C4-PFOA
I13C5-PFNA	13C5-PFNA
I13C2-PFDA	13C2-PFDA
I18O2-PFHxS	18O2-PFHxS
I13C4-PFOS	13C4-PFOS

Extraction Standards

Extraction Standard	Internal Standard
E13C4-PFBA	13C3-PFBA
E13C5-PFPeA	13C2-PFHxA
E13C5-PFHxA	
E13C4-PFHpA	
E13C3-HFPO-DA	
E13C8-PFOA	13C4-PFOA
E13C9-PFNA	13C5-PFNA
E13C6-PFDA	13C2-PFDA
E13C7-PFUnA	
E13C2-PFDoA	
E13C2-PFTeDA	
E13C3-PFBS	18O2-PFHxS
E13C3-PFHxS	
E13C2-4:2-FTS	
E13C2-6:2-FTS	
E13C2-8:2-FTS	

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Extraction Standard	Internal Standard
E13C8-PFOS	13C4-PFOS
E13C8-PFOSA	
Ed3-NMeFOSA	
Ed5-NEtFOSA	
Ed3-NMeFOSAA	
Ed5-NEtFOSAA	
Ed7-NMeFOSE	
Ed9-NEtFOSE	

Native PFAS Compounds

Native	Extraction Standard
PFBA	13C4-PFBA
PFPeA	13C5-PFPeA
3:3FTCA	
PFMPA	
PFMBA	
PFHxA	13C5-PFHxA
NFDHA	
5:3FTCA	
7:3FTCA	
PFEESA	
PFHpA	13C4-PFHpA
PFOA	13C8-PFOA
PFNA	13C9-PFNA
PFDA	13C6-PFDA
PFUnA	13C7-PFUnA
PFDoA	13C2-PFDoA
PFTrDA	Avg 13C2-PFTeDA and 13C2-PFDoA
PFTeDA	13C2-PFTeDA
PFBS	13C3-PFBS
PFPeS	13C3-PFHxS
PFHxS	
PFHpS	13C8-PFOS
PFOS	
PFNS	
PFDS	

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Native	Extraction Standard
PFDoS	
4:2-FTS	13C2-4:2-FTS
6:2-FTS	13C2-6:2-FTS
8:2-FTS	13C2-8:2-FTS
PFOSA	13C8-PFOSA
NMeFOSA	D3-NMeFOSA
NEtFOSA	D5-NEtFOSA
NMeFOSAA	D3-NMeFOSAA
NEtFOSAA	D5-N-EtFOSAA
NMeFOSE	D7-NMeFOSE
NEtFOSE	D9-NEtFOSE
HFPO-DA	13C3-HFPO-DA
DONA	
9Cl-PF3ONS	
11Cl-PF3OUdS	

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Acquisition Method

EPA1633_DOD_23JUL06

Mass Spec 12.500 min
Period 12.500 min
-MRM
Integrated Valve
Sciex LC System
Equilibrate
Injection

Mass Spectrometer Method Properties

Period 1:

Scans in Period: 1250
Min. Dwell Time: 3 ms
Max. Dwell Time: 250 ms
Relative Start Time: 0.00 msec
Scheduled Ionization: Off
Experiments in Period: 1
Use target Cycle Time: No
Target Cycle Time: N/A

Period 1 Experiment 1:

Scan Type: MRM (MRM)
Scheduled MRM: Yes
Polarity: Negative
Scan Mode: N/A
Ion Source: Turbo Spray
sMRM Q1Q3 Resolution: No
MRM detection window: 60 sec
Target Scan Time: 0.6000 sec
Resolution Q1: Unit
Resolution Q3: Unit
Intensity Thres.: 0.00 cps
Settling Time: 0.0000 msec
MR Pause: 5.0070 msec
MCA: No
Step Size: 0.00 Da

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.000	172.000	2.92	DP	-40.00	-40.00	13C3-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
216.800	171.900	2.92	DP	-40.00	-40.00	13C4-PFBA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
268.300	223.000	3.41	DP	-40.00	-40.00	13C5-PFPeA
			CE	-14.00	-14.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
266.000	222.000	3.41	DP	-40.00	-40.00	13C3-PFPeA
			CE	-14.00	-14.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	79.900	3.81	DP	-120.00	-120.00	13C3-PFBS
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	80.900	3.60	DP	-100.00	-100.00	13C2-4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	270.000	3.71	DP	-30.00	-30.00	13C2-PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	273.000	3.71	DP	-40.00	-40.00	13C5-PFHxA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	168.900	3.82	DP	-20.00	-20.00	13C3-HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
367.100	322.000	3.97	DP	-40.00	-40.00	13C4-PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	79.900	4.35	DP	-100.00	-100.00	13C3-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
359.000	294.000	4.60	DP	-40.00	-40.00	13C2-6:2 FTUCA
			CE	-25.00	-25.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
379.000	294.000	4.60	DP	-30.00	-30.00	13C2-6:2 FTCA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	80.900	4.08	DP	-100.00	-100.00	13C2-6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
415.000	370.000	4.20	DP	-50.00	-50.00	13C2-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
417.100	172.000	4.20	DP	-50.00	-50.00	13C4-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
421.100	376.000	4.20	DP	-50.00	-50.00	13C8-PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	79.900	4.93	DP	-100.00	-100.00	13C4-PFOS
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	79.900	4.93	DP	-100.00	-100.00	13C8-PFOS
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
472.100	427.000	4.43	DP	-50.00	-50.00	13C9-PFNA
			CE	-18.00	-18.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
459.000	394.000	5.13	DP	-50.00	-50.00	13C2-8:2 FTUCA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
479.000	394.000	5.13	DP	-35.00	-35.00	13C2-8:2 FTCA
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
519.100	474.100	4.70	DP	-50.00	-50.00	13C6-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.100	470.100	4.70	DP	-50.00	-50.00	13C2-PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	80.900	4.53	DP	-100.00	-100.00	13C2-8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
506.100	77.800	5.78	DP	-100.00	-100.00	13C8-PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
573.200	419.000	4.64	DP	-80.00	-80.00	d3-NMeFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
565.000	520.000	5.07	DP	-70.00	-70.00	13C2-PFUnDA
			CE	-19.00	-19.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.000	525.100	5.07	DP	-70.00	-70.00	13C7-PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
589.200	419.000	4.78	DP	-90.00	-90.00	d5-NEtFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
615.100	570.000	5.55	DP	-60.00	-60.00	13C2-PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
623.200	58.900	6.62	DP	-50.00	-50.00	d7-NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
515.000	219.000	6.76	DP	-100.00	-100.00	d3-NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
639.200	58.900	6.90	DP	-45.00	-45.00	d9-NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
531.100	219.000	7.13	DP	-100.00	-100.00	d5-NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
715.200	670.000	6.40	DP	-60.00	-60.00	13C2-PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

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212.800 168.900 2.92    DP      -40.00 -40.00 PFBA
                                CE      -14.00 -14.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
249.000       99.000        3.12      DP     -60.00 -60.00     PFPrS
                                      CE     -40.00-40.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
229.000       84.900        3.18      DP     -40.00 -40.00     PFECA F
                                      CE     -25.00-25.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
241.000       177.000       3.16      DP     -60.00 -60.00     3:3 FTCA
                                      CE     -12.00-12.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
263.000       219.000       3.41      DP     -40.00 -40.00     PFPeA
                                      CE     -14.00-14.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
298.700       79.900        3.80      DP     -120.00-120.00 PFBS
                                      CE     -65.00-65.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
279.000       85.100        3.51      DP     -40.00 -40.00     PFECA A
                                      CE     -20.00-20.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
314.800       134.900       3.95      DP     -60.00 -60.00  PFEESA
                                      CE     -30.00-30.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
295.000       201.000       3.68      DP     -70.00 -70.00     PFECA B
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				CE	-25.00	-25.00

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	307.000	3.60	DP	-100.00	-100.00	4:2-FTS
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	269.000	3.71	DP	-30.00	-30.00	PFHxA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	79.900	4.09	DP	-90.00	-90.00	PFPeS
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	168.900	3.82	DP	-20.00	-20.00	HFPODA
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	319.000	3.95	DP	-40.00	-40.00	PFHpA
			CE	-15.00	-15.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	79.900	4.35	DP	-100.00	-100.00	PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	250.900	4.04	DP	-40.00	-40.00	DONA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	237.100	3.75	DP	-70.00	-70.00	5:3 FTCA
			CE	-20.00	-20.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
461.000	381.000	4.80	DP	-70.00	-70.00	PFECHS
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	407.000	4.07	DP	-100.00	-100.00	6:2-FTS
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	79.900	4.60	DP	-100.00	-100.00	PFHpS
			CE	-90.00	-90.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	369.000	4.20	DP	-50.00	-50.00	PFOA
			CE	-16.00	-16.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	79.900	4.93	DP	-100.00	-100.00	PFOS
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
463.000	419.000	4.44	DP	-50.00	-50.00	PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	316.900	4.23	DP	-80.00	-80.00	7:3 FTCA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
530.800	351.000	5.28	DP	-100.00	-100.00	9Cl-PF3ONS
			CE	-38.00	-38.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
548.800	79.900	5.39	DP	-100.00	-100.00	PFNS
			CE	-110.00	-110.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
512.900	469.000	4.71	DP	-50.00	-50.00	PFDA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
527.100	507.000	4.53	DP	-100.00	-100.00	8:2-FTS
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	77.900	5.81	DP	-100.00	-100.00	PFOSA
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
570.100	419.000	4.64	DP	-80.00	-80.00	NMeFOSAA
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
599.000	79.900	5.86	DP	-100.00	-100.00	PFDS
			CE	-120.00	-120.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
563.100	519.000	5.08	DP	-70.00	-70.00	PFUnDA
			CE	-19.00	-19.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
584.200	419.100	4.77	DP	-90.00	-90.00	NEtFOSAA
			CE	-30.00	-30.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.900	450.900	6.18	DP	-100.00	-100.00	11Cl-PF3OUdS
			CE	-43.00	-43.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
613.100	569.000	5.55	DP	-60.00	-60.00	PFDoDA
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
616.100	58.900	6.64	DP	-50.00	-50.00	NMePFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	219.000	6.76	DP	-100.00	-100.00	NMePFOSA
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	79.900	6.62	DP	-100.00	-100.00	PFDoS
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
630.000	58.900	6.93	DP	-45.00	-45.00	NEtPFOSAE
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	219.000	7.16	DP	-100.00	-100.00	NEtPFOSA
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	619.000	6.01	DP	-60.00	-60.00	PFTrDA
			CE	-21.00	-21.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	669.000	6.40	DP	-60.00	-60.00	PFTeDA
			CE	-22.00	-22.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
298.700	98.800	3.80	DP	-100.00	-100.00	PFBS_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
295.000	84.900	3.68	DP	-30.00	-30.00	PFECA B_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
327.100	80.900	3.60	DP	-100.00	-100.00	4:2 FTS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
313.000	118.900	3.71	DP	-50.00	-50.00	PFHxA_2
			CE	-31.00	-31.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
349.100	98.900	4.09	DP	-100.00	-100.00	PFPeS_2
			CE	-50.00	-50.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
284.900	184.900	3.82	DP	-75.00	-75.00	HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
385.000	185.000	3.82	DP	-75.00	-75.00	HFPODA_3
			CE	-10.00	-10.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
363.100	169.000	3.95	DP	-60.00	-60.00	PFHpA_2
			CE	-25.00	-25.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
398.700	98.900	4.35	DP	-100.00	-100.00	PFHxS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
341.000	217.000	3.75	DP	-80.00	-80.00	5:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
461.000	99.000	4.80	DP	-60.00	-60.00	PFECHS_2
			CE	-60.00	-60.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
427.100	80.900	4.07	DP	-120.00	-120.00	6:2 FTS_2
			CE	-70.00	-70.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
449.000	98.800	4.60	DP	-100.00	-100.00	PFHpS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
413.000	169.000	4.20	DP	-60.00	-60.00	PFOA_2
			CE	-26.00	-26.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.900	98.800	4.93	DP	-100.00	-100.00	PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID

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463.000 219.000 4.44    DP      -60.00 -60.00 PFNA_2
                                CE      -30.00 -30.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
548.800       98.800        5.39      DP     -100.00-100.00 PFNS_2
                                      CE     -90.00-90.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
512.900       219.000       4.71      DP     -50.00 -50.00  PFDA_2
                                      CE     -31.00-31.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
527.100       80.800        4.53      DP     -100.00-100.00   8:2 FTS_2
                                      CE     -80.00-80.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
570.100       483.000       4.64      DP     -80.00 -80.00    NMeFOSAA_2
                                      CE     -24.00-24.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
599.000       98.800        5.86      DP     -100.00-100.00   PFDS_2
                                      CE     -100.00  -100.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
563.100       269.100       5.08      DP     -80.00 -80.00    PFUnDA_2
                                      CE     -35.00-35.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
584.200       526.000       4.77      DP     -100.00-100.00   NEtFOSAA_2
                                      CE     -30.00-30.00



Q1 Mass (Da)  Q3 Mass (Da)  RT (min)  Param  Start  Stop  ID
613.100       319.000       5.55      DP     -60.00 -60.00    PFDoDA_2
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			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
663.000	168.900	6.01	DP	-60.00	-60.00	PFTrDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
713.100	168.900	6.40	DP	-60.00	-60.00	PFTeDA_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
468.000	423.000	4.42	DP	-50.00	-50.00	13C5-PFNA
			CE	-18.00	-18.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
403.000	83.900	4.33	DP	-100.00	-100.00	18O2-PFHxS
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
263.000	68.900	3.41	DP	-60.00	-60.00	PFPeA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	478.000	5.81	DP	-130.00	-130.00	PFOSA_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
511.900	169.000	6.76	DP	-100.00	-100.00	NMePFOSA_2
			CE	-37.00	-37.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
526.000	169.000	7.16	DP	-180.00	-180.00	NEtPFOSA_2
			CE	-40.00	-40.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
376.900	84.800	4.04	DP	-40.00	-40.00	DONA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
532.800	353.000	5.28	DP	-100.00	-100.00	9Cl-PF3ONS_2
			CE	-38.00	-38.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
632.900	452.900	6.18	DP	-180.00	-180.00	11Cl-PF3OUdS_2
			CE	-40.00	-40.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
241.000	117.000	3.16	DP	-50.00	-50.00	3:3 FTCA_2
			CE	-45.00	-45.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
441.000	336.900	4.23	DP	-80.00	-80.00	7:3 FTCA_2
			CE	-20.00	-20.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
314.800	82.900	3.95	DP	-60.00	-60.00	PFEESA_2
			CE	-30.00	-30.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
699.100	98.800	6.62	DP	-100.00	-100.00	PFDoS_2
			CE	-150.00	-150.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
318.000	120.300	3.71	DP	-180.00	-180.00	13C5-PFHxA_2
			CE	-40.00	-40.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
302.100	98.900	3.81	DP	-120.00	-120.00	13C3-PFBS_2
			CE	-65.00	-65.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
402.100	98.800	4.35	DP	-100.00	-100.00	13C3-PFHxS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
507.100	98.900	4.93	DP	-100.00	-100.00	13C8-PFOS_2
			CE	-80.00	-80.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
329.100	309.000	3.60	DP	-100.00	-100.00	13C2-4:2-FTS_2
			CE	-28.00	-28.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
429.100	409.000	4.08	DP	-100.00	-100.00	13C2-6:2-FTS_2
			CE	-35.00	-35.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
529.100	509.000	4.53	DP	-100.00	-100.00	13C2-8:2-FTS_2
			CE	-42.00	-42.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
286.900	184.900	3.82	DP	-20.00	-20.00	13C3-HFPODA_2
			CE	-10.00	-10.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
315.100	119.400	3.71	DP	-30.00	-30.00	13C2-PFHxA_2
			CE	-18.00	-18.00	

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Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
502.800	98.900	4.93	DP	-100.00	-100.00	13C4-PFOS_2
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.000	80.000	3.62	DP	-100.00	-100.00	TDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.100	80.000	3.86	DP	-100.00	-100.00	TCDCA
			CE	-100.00	-100.00	

Q1 Mass (Da)	Q3 Mass (Da)	RT (min)	Param	Start	Stop	ID
498.200	80.000	3.94	DP	-100.00	-100.00	TUDCA
			CE	-100.00	-100.00	

Parameter Table(Period 1 Experiment 1):

CUR:	35.00
CAD:	10.00
IS:	-3000.00
TEM:	450.00
GS1:	40.00
GS2:	50.00
EP	-10.00
CXP	-14.00

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CERTIFICATE OF ANALYSIS
DOCUMENTATION

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PFAC-MXC

Native Perfluorinated Compound Solution/Mixture

PRODUCT CODE:	PFAC-MXC
LOT NUMBER:	PFACMXC0617
SOLVENT(S):	Methanol / Water (<1%)
DATE PREPARED: (mm/ddlyyyy)	06/14/2017
LAST TESTED: (mm/dd/yyyy)	03/19/2019
EXPIRY DATE: (mmldd/yyyy)	03/19/2024
RECOMMENDED STORAGE:	Store ampoule in a cool, dark place

DESCRIPTION:

PFAC-MXC is a solution/mixture of thirteen native perfluoroalkylcarboxylic acids (C_4-C_{14} C, $_6$ and C $_)$ and eight native perfluoroalkylsulfonates (C.-C $_1$ and C,J The full name, abbreviation and concentration for each of the components are given in Table A.

The individual perfluoroalkylcarboxylic acids and perfluoroalkylsulfonates all have chemical purities of >98%.

DOCUMENTATION/ DATA ATTACHED:

Table A: Components and Concentrations of the Solution/Mixture
Figure 1: LC/MS Data (SIR)
Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

See page 2 for further details.
Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acids to their respective methyl esters.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Wellington Laboratories Inc., 345 Southgate Dr. Guelph ON N1G 3M5 CANADA
519-822-2436 • Fax: 519-822-2849 • info@well-labs.com

INTENDED USE:

The products prepared by Wellington Laboratories Inc. are for laboratory use only. This certified reference material (CRM) was designed to be used as a standard for the identification and/or quantification of the specific chemical compounds it contains.

HANDLING:

This product should only be used by qualified personnel familiar with its potential hazards and trained in the handling of hazardous chemicals. Due care should be exercised to prevent unnecessary human contact or ingestion. All procedures should be carried out in a well-functioning fume hood and suitable gloves, eye protection, and clothing should be worn at all times. Waste should be disposed of according to national and regional regulations. Safety Data Sheets (SDSs) are available upon request.

SYNTHESIS / CHARACTERIZATION:

Our products are synthesized using single-product unambiguous routes whenever possible. They are then characterized, and their structures and purities confirmed, using a combination of the most relevant techniques, such as NMR, GC/MS, LC/MS/MS, SFC/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This further confirms the homogeneity of the crystalline material as well as the stability and homogeneity of the solutions in the storage containers. In order to maintain the integrity of the assigned value(s), and associated uncertainty, the dilution or injection of a subsample of this product should be performed using calibrated measuring equipment.

UNCERTAINTY:

The maximum combined relative standard uncertainty of our reference standard solutions is calculated using the following equation:

The combined relative standard uncertainty, uc(y), of a value y and the uncertainty of the independent parameters $x_1, x_2 \ldots x_n$ on which it depends is:

$$ur(y(xi,x_1, \ldots x_n)) = \sqrt{\sum_{i=1} u(y,x_i)^2}$$

where x is expressed as a relative standard uncertainty of the individual parameter.

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of ±5% (calculated with a coverage factor of 2 and a level of confidence of 95%) is stated on the Certificate of Analysis for all of our products.

TRACEABILITY:

All reference standard solutions are traceable to specific crystalline lots. The microbalances used for solution preparation are regularly calibrated by an external ISO/IEC 17025 accredited laboratory. In addition, their calibration is verified prior to each weighing using calibrated external weights traceable to an ISO/IEC 17025 accredited laboratory. All volumetric glassware used is calibrated, of Class A tolerance, and traceable to an ISO/IEC 17025 accredited laboratory. For certain products, traceability to international interlaboratory studies has also been established.

EXPIRY DATE / PERIOD OF VALIDITY:

Ongoing stability studies of this product have demonstrated stability in its composition and concentration, until the specified expiry date, in the unopened ampoule. Monitoring for any degradation or change in concentration of the listed analyte(s) is performed on a routine basis.

LIMITED WARRANTY:

At the time of shipment, all products are warranted to be free of defects in material and workmanship and to conform to the stated technical and purity specifications.

QUALITY MANAGEMENT:

This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAi Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A 1226), and ISO 17034 by ANSI-ASQ National Accreditation Board (ANAB; AR-1523).

CALA

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REFERENCE MATERIAL PRODUCER

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Table A: **PFAC-MXC; Components and Concentrations (ng/ml, ± 5% in Methanol/ Water (<1%))**

Compound	Abbreviation	Concentration (ng/ml)*		Peak Assignment in Figure 1
Perfluoro-n-butanoic acid	PFBA	2000		A
Perfluoro-n-pentanoic acid	PFPeA	2000		B
Perfluoro-n-hexanoic acid	PFHxA	2000		D
Perfluoro-n-heptanoic acid	PFHpA	2000		F
Perfluoro-n-octanoic acid	PFOA	2000		**H**
Perfluoro-n-nonanoic acid	PFNA	2000		J
Perfluoro-n-decanoic acid	PFDA	2000		L
Perfluoro-n-undecanoic acid	PFUdA	2000		**N**
Perfluoro-n-dodecanoic acid	PFDoA	2000		P
Perfluoro-n-tridecanoic acid	PFTrDA	2000		Q
Perfluoro-n-tetradecanoic acid	PFTeDA	2000		**S**
Perfluoro-n-hexadecanoic acid	PFHxDA	2000		**T**
Perfluoro-n-octadecanoic acid	PFODA	2000		**U**
Compound	**Abbreviation**	**Concentration (ng/ml)***		**Peak Assignment in Figure 1**
		As the salt	**As the anion**	
Potassium perfluoro-1-butanesulfonate	L-PFBS	2000	1770	C
Sodium perfluoro-1-pentanesulfonate	L-PFPeS	2000	1880	E
Sodium perfluoro-1-hexanesulfonate	L-PFHxS	2000	1890	G
Sodium perfluoro-1-heptanesulfonate	L-PFHpS	2000	1900	I
Sodium perfluoro-1-octanesulfonate	L-PFOS	2000	1910	K
Sodium perfluoro-1-nonanesulfonate	L-PFNS	2000	1920	**M**
Sodium perfluoro-1-decanesulfonate	L-PFDS	2000	1930	0
Sodium perfluoro-1-dodecanesulfonate	L-PFDoS	2000	1940	R

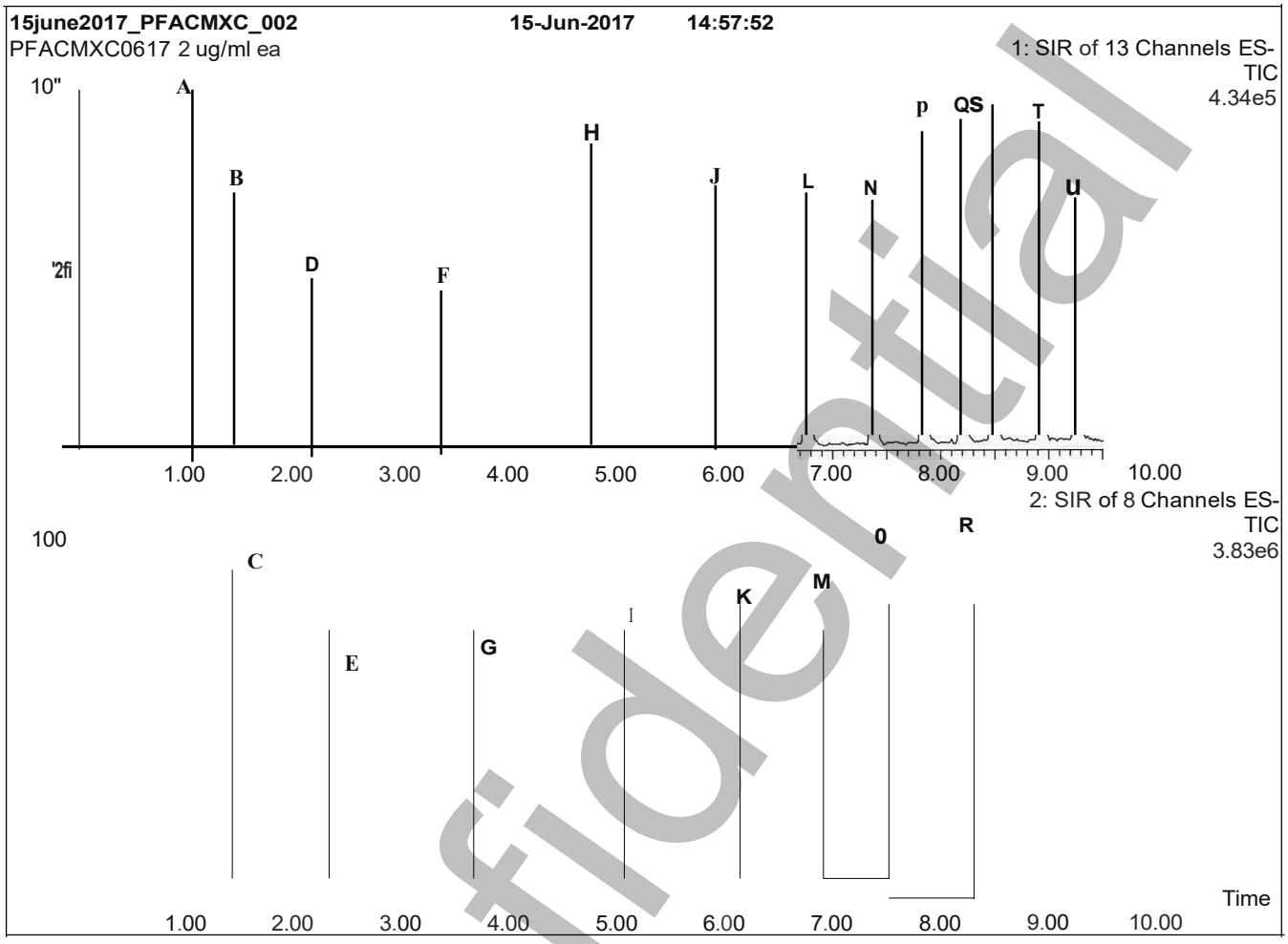
* Concentrations have been rounded to three significant figures.

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Certified By: _____ **Date:** 06/06/2019
(mm/dd/yyyy)

B.G. Chittim, General Manager

Fi.9yr!L1 **PFAC-MXC; LC/MS Data (Total Ion Current Chromatogram; SIR)**



Conditions for Figure 1:

LC: **Waters Acquity Ultra Performance LC**
MS: **Micromass Quattro *micro* API MS**

Chromatographic Conditions

Column: Acquity UPLC BEH Shield RP.,
1.7 µm, 2.1 x 100 mm

Mobile phase: Gradient
Start: 50% H,O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH,OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions in 1 min.

Time: 12 min

Flow: 300 µl/min

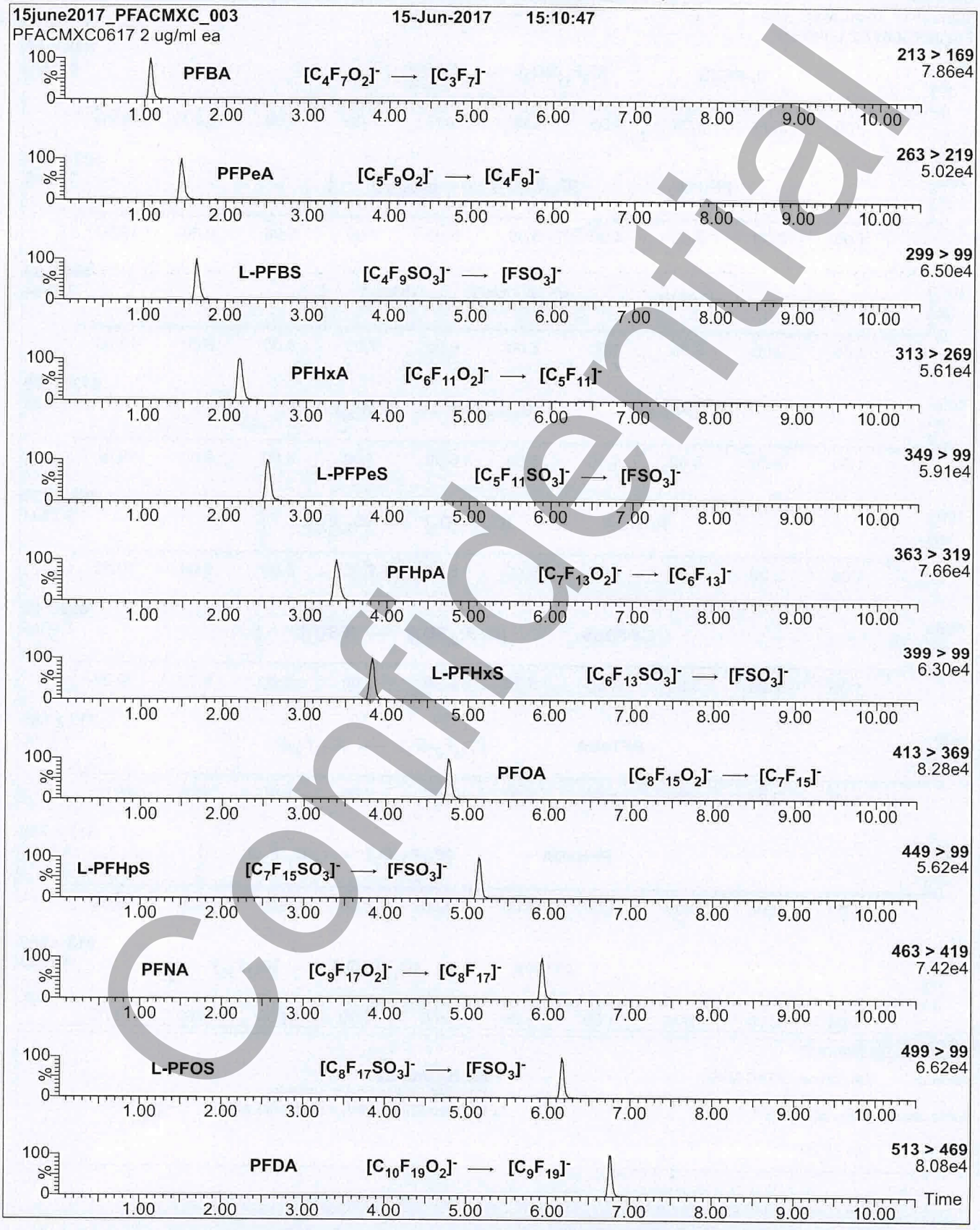
MS Parameters

Experiment: SIR of 21 Channels

Source: Electrospray (negative)
Capillary Voltage (kV) = 3.00
Cone Voltage (V) = variable (10-80)
Cone Gas Flow (l/hr) = 50
Desolvation Gas Flow (l/hr) = 750

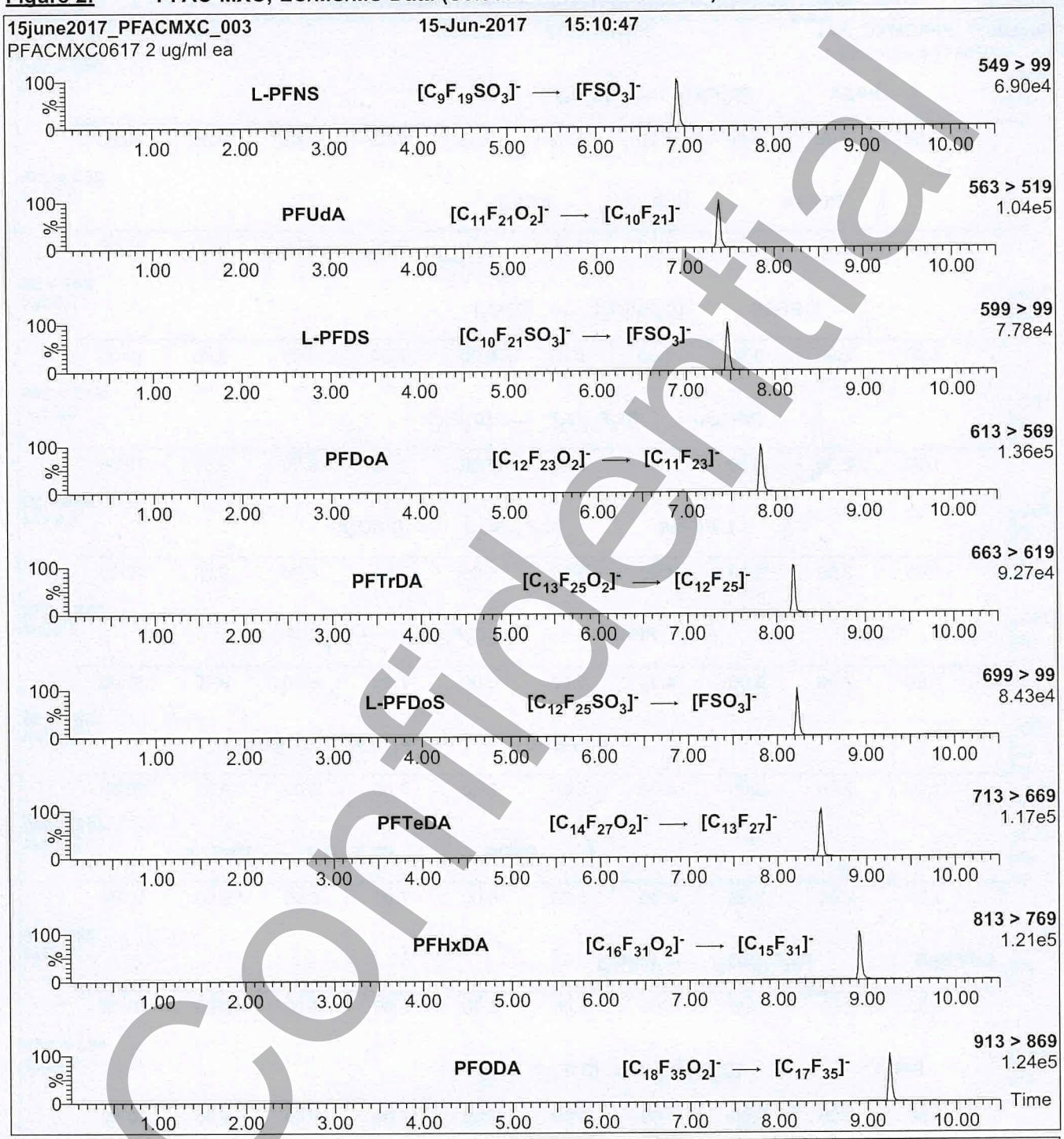
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Figure 2: **PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)**



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Figure 2: **PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)**



Conditions for Figure 2:

Injection: On-column (PFAC-MXC)

Mobile phase: Same as Figure 1

Flow: 300 µl/min

MS Parameters
Collision Gas (mbar) = 3.46e-3
Collision Energy (eV) = 8-50 (variable)

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Native PFAS Intermediate A								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Native PFAS Intermediate (ng/ml)**
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.100		471.0
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.100		466.0
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	47100	0.100		471.0
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	50000	0.100		500.0
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	47900	0.100		479.0
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	46700	0.100		467.0
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	47400	0.100		474.0
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.025		125.0
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.025		125.0
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.025		110.5
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.100		500.0
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.025		120.5
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.025		125.0
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	48400	0.025		121.0
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	50000	0.025		125.0
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.025		119.0
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.025		125.0
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.025		113.8
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.025	10	125.0
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.025		120.0
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.025		125.0
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.025		125.0
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46400	0.025		116.0
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.025		125.0
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900	0.025		117.3
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.050		250.0
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.025		125.0
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.025		125.0
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	50000	0.025		125.0
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.050		250.0
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	50000	0.050		250.0
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50000	0.050		250.0
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.050		222.5
Wellington	br-NMeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSE	50000	0.250		1250.0
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	50000	0.025		125.0
Wellington	br-NEtFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSE	50000	0.250		1250.0
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	50000	0.025		125.0
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.125		625.0
Wellington	FPePA	3-Perflurorpentylpropanoic acid	914637-49-3	5:3 FTCA	50000	0.625		3125.0
Wellington	FHpPA	3-Perfluoroheptylpropanoic acid	812-70-4	7:3 FTCA	50000	0.625		3125.0

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Native PFAS Intermediate B								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Native PFAS Intermediate (ng/ml)**
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.200		942.0
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.200		932.0
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	47100	0.200		942.0
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	50000	0.200		1000.0
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	47900	0.200		958.0
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	46700	0.200		934.0
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	47400	0.200		948.0
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.050		250.0
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.050		250.0
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.050		221.0
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.200		1000.0
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.050		241.0
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.050		250.0
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	48400	0.050		242.0
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	50000	0.050		250.0
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.050		238.0
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.050		250.0
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.050	10	227.5
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.050		250.0
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.050		240.0
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.050		250.0
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.050		250.0
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46400	0.050		232.0
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.050		250.0
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900	0.050		234.5
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.100		500.0
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.050		250.0
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.050		250.0
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	50000	0.050		250.0
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.100		500.0
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	50000	0.100		500.0
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50000	0.100		500.0
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.100		445.0
Wellington	br-NMeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSAE	50000	0.500		2500.0
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	50000	0.050		250.0
Wellington	br-NEtFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSAE	50000	0.500		2500.0
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	50000	0.050		250.0
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.25		1250.0

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1633 Extracted-Internal Standard (EIS) Intermediate								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL of MeOH)**	**Final Conc. Native PFAS Intermediate (ng/ml)**
Wellington	MPFBA	Perfluoro-n-($^{13}C_4$)butanoic acid	STL00992	$^{13}C_4$-PFBA	50000	0.4		2000.0
Wellington	M5PFPeA	Perfluoro-n-($^{13}C_5$)pentanoic acid	STL01893	$^{13}C_5$-PFBA	50000	0.2		1000.0
Wellington	M5PFHxA	Perfluoro-n-(1,2,3,4,6-$^{13}C_5$)hexanoic acid	STL02577	$^{13}C_5$-PFHxA	50000	0.1		500.0
Wellington	M4PFHpA	Perfluoro-n-(1,2,3,4-$^{13}C_4$)heptanoic acid	STL01892	$^{13}C_4$-PFHpA	50000	0.1		500.0
Wellington	M8PFOA	Perfluoro-n-($^{13}C_8$)octanoic acid	STL01052	$^{13}C_8$-PFOA	50000	0.1		500.0
Wellington	M9PFNA	Perfluoro-n-($^{13}C_9$)nonanoic acid	STL02578	$^{13}C_9$-PFNA	50000	0.05		250.0
Wellington	M6PFDA	Perfluoro-n-(1,2,3,4,5,6-$^{13}C_6$)decanoic acid	STL02579	$^{13}C_6$-PFDA	50000	0.05		250.0
Wellington	M7PFUdA	Perfluoro-n-(1,2,3,4,5,6,7-$^{13}C_7$)undecanoic acid	STL02580	$^{13}C_7$-PFUnA	50000	0.05		250.0
Wellington	MPFDoA	Perfluoro-n-(1,2-$^{13}C_2$)dodecanoic acid	STL02703	$^{13}C_2$-PFDoDA	50000	0.05		250.0
Wellington	M2PFTeDA	Perfluoro-n-(1,2-$^{13}C_2$)tetradecanoic acid	STL02116	$^{13}C_2$-PFTeDA	50000	0.05		250.0
Wellington	M3PFBS	Perfluoro-1-(2,3,4-$^{13}C_3$)butanesulfonic acid	STL02337	$^{13}C_3$-PFBS	46500	0.1		465.0
Wellington	M3PFHxS	Perfluoro-1-(1,2,3-$^{13}C_3$)hexanesulfonic acid	STL02581	$^{13}C_3$-PFHxS	47300	0.1		473.0
Wellington	M8PFOS	Perfluoro-1-($^{13}C_8$)octanesulfonic acid	STL01054	$^{13}C_8$-PFOS	47800	0.1	10	478.0
Wellington	M8FOSA	Perfluoro-1-($^{13}C_8$)octanesulfonamide	STL01056	$^{13}C_8$-FOSA	50000	0.1		500.0
Wellington	d3-N-MeFOSAA	N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	d_3-NMeFOSAA	50000	0.2		1000.0
Wellington	d5-N-EtFOSAA	N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	d_5-NEtFOSAA	50000	0.2		1000.0
Wellington	M2-4:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)hexanesulfonic Acid	STL02395	$^{13}C_2$-4:2FTS	46700	0.2		934.0
Wellington	M2-6:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)octanesulfonic Acid	STL02279	$^{13}C_2$-6:2FTS	47500	0.2		950.0
Wellington	M2-8:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)decanesulfonic Acid	STL02280	$^{13}C_2$-8:2FTS	47900	0.2		958.0
Wellington	M3HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)($^{13}C_3$)propanoic acid	STL02255	$^{13}C_3$-HFPO-DA	50000	0.4		2000.0
Wellington	d7-N-MeFOSE	2-(N-methyl-d_3-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02277	d_7-NMeFOSE	50000	1		5000.0
Wellington	d9-N-EtFOSE	2-(N-ethyl-d_5-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02278	d_9-NEtFOSE	50000	1		5000.0
Wellington	d-N-MeFOSA	N-ethyl-d_5-perfluoro-1-octanesulfonamide	STL02704	d_5-NEtFOSA	50000	0.1		500.0
Wellington	d-N-EtFOSA	N-methyl-d_3-perfluoro-1-octanesulfonamide	STL02705	d_3-NMeFOSA	50000	0.1		500.0

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1633 Non-extracted Internal Standard (NIS) Intermediate								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Native PFAS Intermediate (ng/ml)**
Wellington	M3PFBA	Perfluoro-n-(2,3,4-$^{13}C_3$)butanoic acid	STL02680	$^{13}C_3$-PFBA	50000	0.20		1000.0
Wellington	MPFOA	Perfluoro-n-(1,2,3,4-$^{13}C_4$)octanoic acid	STL00990	$^{13}C_4$-PFOA	50000	0.10		500.0
Wellington	MPFDA	Perfluoro-n-(1,2-$^{13}C_2$)decanoic acid	STL00996	$^{13}C_2$-PFDA	50000	0.05		250.0
Wellington	MPFOS	Perfluoro-1-(1,2,3,4-$^{13}C_4$)octanesulfonic Acid	STL00991	$^{13}C_4$-PFOS	47800	0.10	10	478.0
Wellington	MPFNA	Perfluoro-n-(1,2,3,4,5-$^{13}C_5$)nonanoic acid	STL00995	$^{13}C_5$-PFNA	50000	0.05		250.0
Wellington	MPFHxA	Perfluoro-n-(1,2-$^{13}C_2$)hexanoic acid	STL00993	$^{13}C_2$-PFHxA	50000	0.10		500.0
Wellington	MPFHxS	Perfluoro-1-hexane($^{18}O_2$)sulfonic acid	STL00994	$^{18}O_2$-PFHxS	47300	0.10		473.0

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1633 TOP SS Intermediate								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc 1633 TOP Intermediate (ng/ml)**
Wellington	M2PFOA	Perfluoro-n-(1,2-$^{13}C_2$)octanoic acid	STL00623	$^{13}C_2$-PFOA	50000	0.020	2	500
Wellington	MPFUdA	Perfluoro-n-(1,2-$^{13}C_2$)undecanoic acid	STL00997	$^{13}C_2$-PFUnA	50000	0.010		250
Wellington	M3PFPeA	Perfluoro-n-(3,4,5-$^{13}C_3$)pentanoic acid	STL03269	^{13}C3-PFPeA	50000	0.040		1000

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1633 Initial Calibration Standards Preparation							
Solution Name	**CAL1**	**CAL2**	**CAL3**	**CAL4**	**CAL5**	**CAL6**	**CAL7**
Native PFAS Intermediate A (mL)	0.016	0.040	0.100	0.200	0.400	NA	NA
Native PFAS Intermediate B (mL)	NA	NA	NA	NA	NA	0.500	2.500
1633 TOP SS Intermediate (mL)	0.050	0.050	0.050	0.050	0.050	0.050	0.050
1633 EIS Intermediate (mL)	0.050	0.050	0.050	0.050	0.050	0.050	0.050
1633 NIS Intermediate (mL)	0.050	0.050	0.050	0.050	0.050	0.050	0.050
Native FPePA Stock (5:3 FTCA) (mL)	NA	NA	NA	NA	NA	0.062	0.312
Native FHpPA Stock (7:3 FTCA) (mL)	NA	NA	NA	NA	NA	0.062	0.312
Final Volume (mL) (methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid)	10	10	10	10	10	10	10

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1633 Initial Calibration Standards Concentrations							
	CAL1	CAL2	CAL3	CAL4	CAL5	CAL6	CAL7
Compound Name	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)	Conc. (ng/ml)
PFBA	0.8	2	5	10	20	50	250
PFPeA	0.4	1	2.5	5	10	25	125
PFHxA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFHpA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFOA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFNA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFUnA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFDoA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFTrDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFTeDA	0.2	0.5	1.25	2.5	5	12.5	62.5
PFBS	0.1768	0.442	1.105	2.21	4.42	11.05	55.25
PFPeS	0.1876	0.469	1.1725	2.345	4.69	11.725	58.625
PFHxS	0.182	0.455	1.1375	2.275	4.55	11.375	56.875
PFHpS	0.1904	0.476	1.19	2.38	4.76	11.05	55.25
PFOS	0.1856	0.464	1.16	2.32	4.64	11.6	58
PFNS	0.192	0.48	1.2	2.4	4.8	12	60
PFDS	0.1928	0.482	1.205	2.41	4.82	12.05	60.25
PFDoS	0.1936	0.484	1.21	2.42	4.84	12.1	60.5
8:2FTS	0.7664	1.916	4.79	9.58	19.16	47.9	239.5
4:2FTS	0.7472	1.868	4.67	9.34	18.68	46.7	233.5
6:2FTS	0.7584	1.896	4.74	9.48	18.96	47.4	237
PFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NEtFOSA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSAA	0.2	0.5	1.25	2.5	5	12.5	62.5
NEtFOSAA	0.2	0.5	1.25	2.5	5	12.5	62.5
NMeFOSE	2	5	12.5	25	50	125	625
NEtFOSE	2	5	12.5	25	50	125	625
HFPO-DA	0.8	2	5	10	20	50	250
ADONA	0.7536	1.884	4.71	9.42	18.84	47.1	235.5
PFMPA	0.4	1	2.5	5	10	25	125
PFMBA	0.4	1	2.5	5	10	25	125
NFDHA	0.4	1	2.5	5	10	25	125
9Cl-PF3ONS	0.7456	1.864	4.66	9.32	18.64	46.6	233
11Cl-PF3OUdS	0.7536	1.884	4.71	9.42	18.84	47.1	235.5
PFEESA	0.356	0.89	2.225	4.45	8.9	22.25	111.25
3:3FTCA	1	2.5	6.25	12.5	25	62.5	312.5
5:3FTCA	5	12.5	31.25	62.5	125	312	1560
7:3FTCA	5	12.5	31.25	62.5	125	312	1560
13C4-PFBA	10	10	10	10	10	10	10
13C5-PFPeA	5	5	5	5	5	5	5
13C5-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C4-PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C8-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C9-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C6-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C7-PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFTeDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C3-PFBS	2.33	2.33	2.33	2.33	2.33	2.33	2.33
13C3-PFHxS	2.37	2.37	2.37	2.37	2.37	2.37	2.37
13C8-PFOS	2.395	2.395	2.395	2.395	2.395	2.395	2.395
13C2-4:2 FTS	4.69	4.69	4.69	4.69	4.69	4.69	4.69
13C2-6:2 FTS	4.755	4.755	4.755	4.755	4.755	4.755	4.755
13C2-8:2 FTS	4.8	4.8	4.8	4.8	4.8	4.8	4.8
13C8-PFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D3-NMeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D5-NEtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D3-NMeFOSAA	5	5	5	5	5	5	5
D5-NEtFOSAA	5	5	5	5	5	5	5
D7-NMeFOSE	25	25	25	25	25	25	25
D9-NEtFOSE	25	25	25	25	25	25	25
13C2-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C2-PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C3-PFPeA	5	5	5	5	5	5	5
13C3-HFPO-DA	10	10	10	10	10	10	10
13C3-PFBA	5	5	5	5	5	5	5
13C2-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C4-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C5-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25
18O2-PFHxS	2.37	2.37	2.37	2.37	2.37	2.37	2.37
13C4-PFOS	2.395	2.395	2.395	2.395	2.395	2.395	2.395

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Bile Salt Stock Solution							
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Aliquot (g)**	**Final Volume (mL MeOH)**	**Final Conc. Bile Salt Stock Solution Adjusted for Acid (ng/ml)**
Sigma Alrich	T0557-500MG	Taurodeoxycholic Acid (TDCA)	516-50-7	TDCA	0.05	50	1000000
Sigma Alrich	T6260-1G	Taurochenodeoxycholic Acid (TCDCA)	516-35-8	TCDCA	0.05		950000
Calbiochem	580549-1GM	Tauroursodeoxycholic Acid (TUDCA)	14605-22-2	TUDCA	0.05		1000000

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Bile Salts Intermediate							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (mL)**	**Final Volume (mL) Methanol**	**Final Conc. Bile Salt Intermediate Solution (ng/ml)**
Bile Salts Stock Solution	Taurodeoxycholic Acid (TDCA)	516-50-7	TDCA	1000000	1	4	250000
	Taurochenodeoxycholic Acid (TCDCA)	516-35-8	TCDCA	950000			237500
	Tauroursodeoxycholic Acid (TUDCA)	14605-22-2	TUDCA	1000000			250000

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Bile Salts Working Solution							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Bile Salts Working Solution (ng/ml)**
Bile Salts Intermediate	Taurodeoxycholic Acid (TDCA)	516-50-7	TDCA	250000	0.10	5	5000
	Taurochenodeoxycholic Acid (TCDCA)	516-35-8	TCDCA	237500			4750
	Tauroursodeoxycholic Acid (TUDCA)	14605-22-2	TUDCA	250000			5000

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1633 Window-Defining Matrix Standard							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume* (ml)**	**Final Conc. 1633 WDM Solution (ng/ml)**
Bile Salt Working Solution	Taurodeoxycholic Acid (TDCA)	516-50-7	TDCA	5000	0.20	10	100
	Taurochenodeoxycholic Acid (TCDCA)	516-35-8	TCDCA	4750			95
	Tauroursodeoxycholic Acid (TUDCA)	14605-22-2	TUDCA	5000			100
1633 TOP SS Intermediate	Perfluoro-n-(1,2-$^{13}C_2$)octanoic acid	STL00623	$^{13}C_2$-PFOA	500	0.05		2.500
	Perfluoro-n-(1,2-$^{13}C_2$)undecanoic acid	STL00997	$^{13}C_2$-PFUnA	250			1.250
	Perfluoro-n-(3,4,5-$^{13}C_3$)pentanoic acid	STL03269	^{13}C3-PFPeA	1000			5.000
EIS Intermediate	Perfluoro-n-($^{13}C_4$)butanoic acid	STL00992	$^{13}C_4$-PFBA	2000	0.05		10.000
	Perfluoro-n-($^{13}C_5$)pentanoic acid	STL01893	$^{13}C_5$-PFBA	1000			5.000
	Perfluoro-n-(1,2,3,4,6-$^{13}C_5$)hexanoic acid	STL02577	$^{13}C_5$-PFHxA	500			2.500
	Perfluoro-n-(1,2,3,4-$^{13}C_4$)heptanoic acid	STL01892	$^{13}C_4$-PFHpA	500			2.500
	Perfluoro-n-($^{13}C_8$)octanoic acid	STL01052	$^{13}C_8$-PFOA	500			2.500
	Perfluoro-n-($^{13}C_9$)nonanoic acid	STL02578	$^{13}C_9$-PFNA	250			1.250
	Perfluoro-n-(1,2,3,4,5,6-$^{13}C_6$)decanoic acid	STL02579	$^{13}C_6$-PFDA	250			1.250
	Perfluoro-n-(1,2,3,4,5,6,7-$^{13}C_7$)undecanoic acid	STL02580	$^{13}C_7$-PFUnA	250			1.250
	Perfluoro-n-(1,2-$^{13}C_2$)dodecanoic acid	STL02703	$^{13}C_2$-PFDoDA	250			1.250
	Perfluoro-n-(1,2-$^{13}C_2$)tetradecanoic acid	STL02116	$^{13}C_2$-PFTeDA	250			1.250
	Perfluoro-1-(2,3,4-$^{13}C_3$)butanesulfonic acid	STL02337	$^{13}C_3$-PFBS	465			2.325
	Perfluoro-1-(1,2,3-$^{13}C_3$)hexanesulfonic acid	STL02581	$^{13}C_3$-PFHxS	473			2.365
	Perfluoro-1-($^{13}C_8$)octanesulfonic acid	STL01054	$^{13}C_8$-PFOS	478			2.390
	Perfluoro-1-($^{13}C_8$)octanesulfonamide	STL01056	$^{13}C_8$-FOSA	500			2.500
	N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	d_3-NMeFOSAA	1000			5.000
	N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	d_5-NEtFOSAA	1000			5.000
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)hexanesulfonic Acid	STL02395	$^{13}C_2$-4:2FTS	934			4.670
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)octanesulfonic Acid	STL02279	$^{13}C_2$-6:2FTS	950			4.750
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)decanesulfonic Acid	STL02280	$^{13}C_2$-8:2FTS	958			4.790
	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)($^{13}C_3$)propanoic acid	STL02255	$^{13}C_3$-HFPO-DA	2000			10.000
	2-(N-methyl-d_3-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02277	d_7-NMeFOSE	5000			25.000
	2-(N-ethyl-d_5-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02278	d_9-NEtFOSE	5000			25.000
	N-ethyl-d_5-perfluoro-1-octanesulfonamide	STL02704	d_5-NEtFOSA	500			2.500
	N-methyl-d_3-perfluoro-1-octanesulfonamide	STL02705	d_3-NMeFOSA	500			2.500
NIS Intermediate	Perfluoro-n-(2,3,4-$^{13}C_3$)butanoic acid	STL02680	$^{13}C_3$-PFBA	1000	0.05		5.000
	Perfluoro-n-(1,2,3,4-$^{13}C_4$)octanoic acid	STL00990	$^{13}C_4$-PFOA	500			2.500
	Perfluoro-n-(1,2-$^{13}C_2$)decanoic acid	STL00996	$^{13}C_2$-PFDA	250			1.250
	Perfluoro-1-(1,2,3,4-$^{13}C_4$)octanesulfonic Acid	STL00991	$^{13}C_4$-PFOS	478			2.390
	Perfluoro-n-(1,2,3,4,5-$^{13}C_5$)nonanoic acid	STL00995	$^{13}C_5$-PFNA	250			1.250
	Perfluoro-n-(1,2-$^{13}C_2$)hexanoic acid	STL00993	$^{13}C_2$-PFHxA	500			2.500
	Perfluoro-1-hexane($^{18}O_2$)sulfonic acid	STL00994	$^{18}O_2$-PFHxS	473			2.365

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* Bring to final volume using methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid

Native 1633 Mid-Level Spike								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Native 1633 Mid-Level Spike (ng/ml)**
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.20		942.0
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.20		932.0
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	47100	0.20		942.0
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	50000	0.20		1000.0
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	47900	0.20		958.0
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	46700	0.20		934.0
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	47400	0.20		948.0
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.05		250.0
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.05		250.0
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.05		221.0
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.20		1000.0
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.05		241.0
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.05		250.0
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	48400	0.05		242.00
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	50000	0.05		250.00
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.05		238.00
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.05		250.00
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.05		227.50
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.05		250.00
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.05	10	240.00
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.05		250.00
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.05		250.00
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46400	0.05		232.00
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.05		250.00
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900	0.05		234.50
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.10		500.00
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.05		250.00
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.05		250.00
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	50000	0.05		250.00
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.10		500.00
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	50000	0.10		500.00
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50000	0.10		500.00
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.10		445.00
Wellington	br-NMeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	24448-09-7	NMePFOSE	50000	0.50		2500.00
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	50000	0.05		250.00
Wellington	br-NEtFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	1691-99-2	NEtPFOSE	50000	0.50		2500.00
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	50000	0.05		250.00
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.25		1250.00
Wellington	FPePA	3-Perflurorpentylpropanoic acid	914637-49-3	5:3 FTCA	50000	1.25		6250.00
Wellington	FHpPA	3-Perfluoroheptylpropanoic acid	812-70-4	7:3 FTCA	50000	1.25		6250.00

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Native 1633 Low-Level Intermediate								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Native 1633 Low-Level Spike (ng/ml)**
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100	0.40		1884.0
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600	0.40		1864.0
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	47100	0.40		1884.0
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	50000	0.40		2000.0
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	47900	0.40		1916.0
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	46700	0.40		1868.0
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	47400	0.40		1896.0
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000	0.10		500.0
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000	0.10		500.0
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200	0.10		442.0
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000	0.40		2000.0
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200	0.10		482.0
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000	0.10		500.0
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	48400	0.10		484.0
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	50000	0.10		500.0
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600	0.10		476.0
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000	0.10		500.0
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500	0.10	10	455.0
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000	0.10		500.0
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000	0.10		480.0
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000	0.10		500.0
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000	0.10		500.0
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46400	0.10		464.0
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000	0.10		500.0
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900	0.10		469.0
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000	0.20		1000.0
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000	0.10		500.0
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000	0.10		500.0
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	50000	0.10		500.0
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000	0.20		1000.0
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	50000	0.20		1000.0
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50000	0.20		1000.0
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500	0.20		890.0
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	50000	0.10		500.0
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	50000	0.10		500.0
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000	0.50		2500.0

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Native 1633 Low-Level Spike								
Vendor	**Catalog Number**	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/mL)**	**Aliquot (mL)**	**Final Volume (mL MeOH)**	**Final Conc. Native 1633 Low-Level Spike (ng/ml)**
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	1884			188.40
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	1864			186.40
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	1884			188.40
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	2000			200.00
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	1916			191.60
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	1868			186.80
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	1896			189.60
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	500			50.00
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	500			50.00
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	442			44.20
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	2000			200.00
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	482			48.20
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	500			50.00
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	484			48.40
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	500			50.00
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	476			47.60
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	500			50.00
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	455	1.00		45.50
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	500			50.00
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	480			48.00
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	500		10	50.00
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	500			50.00
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	464			46.40
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	500			50.00
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	469			46.90
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	1000			100.00
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	500			50.00
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	500			50.00
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	500			50.00
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	1000			100.00
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	1000			100.00
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	1000			100.00
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	890			89.00
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	500			50.00
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	500			50.00
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	2500			250.00
Wellington	FPePA	3-Perflurorpentylpropanoic acid	914637-49-3	5:3 FTCA	50000	0.250		1250.00
Wellington	FHpPA	3-Perfluoroheptylpropanoic acid	812-70-4	7:3 FTCA	50000	0.2500		1250.00
Wellington	N-MeFOSE-M	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSAE	50000	0.1000		500.00
Wellington	N-EtFOSE-M	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NEtPFOSAE	50000	0.1000		500.00

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PFAS 1633 ICV Working Standard							
Solution Name	**Analyte**	**CAS#**	**Acronym**	**Conc. (ng/ml)**	**Aliquot (ml)**	**Final Volume* (ml)**	**Final Conc. PFAS 1633 ICV Working Standard (ng/ml)**
ICV Native PFAS Intermediate A	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	471.0	0.2	10	9.42
	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	466.0			9.32
	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	471.0			9.42
	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	500			10.00
	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	479			9.58
	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	467.0			9.34
	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	474			9.48
	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	125			2.50
	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	125			2.50
	Perfluorobutanesulfonic acid	375-73-5	PFBS	110.5			2.21
	Perfluorobutanoic acid	375-22-4	PFBA	500			10.00
	Perfluorodecanesulfonic acid	335-77-3	PFDS	120.5			2.41
	Perfluorodecanoic acid	335-76-2	PFDA	125			2.50
	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	121.0			2.42
	Perfluorododecanoic acid	307-55-1	PFDoDA	125			2.50
	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	119.0			2.38
	Perfluoroheptanoic acid	375-85-9	PFHpA	125			2.50
	Perfluorohexanesulfonic acid	355-46-4	PFHxS	113.8			2.28
	Perfluorohexanoic acid	307-24-4	PFHxA	125			2.50
	Perfluorononanesulfonic acid	68259-12-1	PFNS	120.0			2.40
	Perfluorononanoic acid	375-95-1	PFNA	125			2.50
	Perfluorooctanesulfonamide	754-91-6	PFOSA	125			2.50
	Perfluorooctanesulfonic acid	1763-23-1	PFOS	116.0			2.32
	Perfluorooctanoic acid	335-67-1	PFOA	125			2.50
	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	117.3			2.35
	Perfluoropentanoic acid	2706-90-3	PFPeA	250			5.00
	Perfluorotetradecanoic acid	376-06-7	PFTeDA	125			2.50
	Perfluorotridecanoic acid	72629-94-8	PFTrDA	125			2.50
	Perfluoroundecanoic acid	2058-94-8	PFUnDA	125			2.50
	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	250			5.00
	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	250			5.00
	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	250			5.00
	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	222.5			4.45
	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSE	1250			25.00
	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	125			2.50
	2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	1691-99-2	NEtPFOSE	1250			25.00
	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	125			2.50
	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	625			12.50
5:3 FTCA Stock	3-Perflurorpentylpropanoic acid	914637-49-3	5:3 FTCA	3125	0.0125		3.91
7:3 FTCA Stock	3-Perfluoroheptylpropanoic acid	812-70-4	7:3 FTCA	3125	0.0125		3.91
1633 EIS Intermediate	Perfluoro-n-($^{13}C_4$)butanoic acid	STL00992	$^{13}C_4$-PFBA	2000	0.050		10.00
	Perfluoro-n-($^{13}C_5$)pentanoic acid	STL01893	$^{13}C_5$-PFBA	1000			5.00
	Perfluoro-n-(1,2,3,4,6-$^{13}C_5$)hexanoic acid	STL02577	$^{13}C_5$-PFHxA	500			2.50
	Perfluoro-n-(1,2,3,4-$^{13}C_4$)heptanoic acid	STL01892	$^{13}C_4$-PFHpA	500			2.50
	Perfluoro-n-($^{13}C_8$)octanoic acid	STL01052	$^{13}C_8$-PFOA	500			2.50
	Perfluoro-n-($^{13}C_9$)nonanoic acid	STL02578	$^{13}C_9$-PFNA	250			1.25
	Perfluoro-n-(1,2,3,4,5,6-$^{13}C_6$)decanoic acid	STL02579	$^{13}C_6$-PFDA	250			1.25
	Perfluoro-n-(1,2,3,4,5,6,7-$^{13}C_7$)undecanoic acid	STL02580	$^{13}C_7$-PFUnA	250			1.25
	Perfluoro-n-(1,2-$^{13}C_2$)dodecanoic acid	STL02703	$^{13}C_2$-PFDoDA	250			1.25
	Perfluoro-n-(1,2-$^{13}C_2$)tetradecanoic acid	STL02116	$^{13}C_2$-PFTeDA	250			1.25
	Perfluoro-1-(2,3,4-$^{13}C_3$)butanesulfonic acid	STL02337	$^{13}C_3$-PFBS	465			2.33
	Perfluoro-1-(1,2,3-$^{13}C_3$)hexanesulfonic acid	STL02581	$^{13}C_3$-PFHxS	473			2.37
	Perfluoro-1-($^{13}C_8$)octanesulfonic acid	STL01054	$^{13}C_8$-PFOS	478			2.39
	Perfluoro-1-($^{13}C_8$)octanesulfonamide	STL01056	$^{13}C_8$-FOSA	500			2.50
	N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	d_3-NMeFOSAA	1000			5.00
	N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	d_5-NEtFOSAA	1000			5.00
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)hexanesulfonic Acid	STL02395	$^{13}C_2$-4:2FTS	934			4.67
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)octanesulfonic Acid	STL02279	$^{13}C_2$-6:2FTS	950			4.75
	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)decanesulfonic Acid	STL02280	$^{13}C_2$-8:2FTS	958			4.79
	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)($^{13}C_3$)propanoic acid	STL02255	$^{13}C_3$-HFPO-DA	2000			10.00
	2-(N-methyl-d_3-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02277	d_7-NMeFOSE	5000			25.00
	2-(N-ethyl-d_5-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02278	d_9-NEtFOSE	5000			25.00
	N-ethyl-d_5-perfluoro-1-octanesulfonamide	STL02704	d_5-NEtFOSA	500			2.50
	N-methyl-d_3-perfluoro-1-octanesulfonamide	STL02705	d_3-NMeFOSA	500			2.50
1633 TOP SS Intermediate	Perfluoro-n-(1,2-$^{13}C_2$)octanoic acid	STL00623	$^{13}C_2$-PFOA	500	0.050		2.50
	Perfluoro-n-(1,2-$^{13}C_2$)undecanoic acid	STL00997	$^{13}C_2$-PFUnA	250			1.25
	Perfluoro-n-(3,4,5-$^{13}C_3$)pentanoic acid	STL03269	^{13}C3-PFPeA	1000			5.00
1633 NIS Intermediate	Perfluoro-n-(2,3,4-$^{13}C_3$)butanoic acid	STL02680	$^{13}C_3$-PFBA	1000	0.05		5.00
	Perfluoro-n-(1,2,3,4-$^{13}C_4$)octanoic acid	STL00990	$^{13}C_4$-PFOA	500			2.50
	Perfluoro-n-(1,2-$^{13}C_2$)decanoic acid	STL00996	$^{13}C_2$-PFDA	250			1.25
	Perfluoro-1-(1,2,3,4-$^{13}C_4$)octanesulfonic Acid	STL00991	$^{13}C_4$-PFOS	478			2.39
	Perfluoro-n-(1,2,3,4,5-$^{13}C_5$)nonanoic acid	STL00995	$^{13}C_5$-PFNA	250			1.25
	Perfluoro-n-(1,2-$^{13}C_2$)hexanoic acid	STL00993	$^{13}C_2$-PFHxA	500			2.50
	Perfluoro-1-hexane($^{18}O_2$)sulfonic acid	STL00994	$^{18}O_2$-PFHxS	473			2.37

* Bring to final volume using methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid

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Purchased Standards					
Vendor	Catalog Number	Analyte	CAS#	Acronym	Conc. (ng/mL)
Wellington	PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	44500
Wellington	HFPO-DA	Perfluoro(2-propxypropanoic) acid	13252-13-6	HFPODA	50000
Calbiochem	580549-1GM	Tauroursodeoxycholic Acid (TUDCA)	14605-22-2	TUDCA	neat
Wellington	3,6-OPFHpA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6	NFDHA	50000
Wellington	br-NEtFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	1691-99-2	NEtPFOSE	50000
Wellington	br-NEtFOSE	2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol	1691-99-2	NEtPFOSAE	50000
Wellington	br-PFOSK	Perfluorooctanesulfonic acid	1763-23-1	PFOS	46400
Wellington	PFUdA	Perfluoroundecanoic acid	2058-94-8	PFUnDA	50000
Wellington	br-NMeFOSAA	N-methylperfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	50000
Wellington	br-NMeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSE	50000
Wellington	br-NMeFOSE	2-(N-methylperfluoro-1-octanesulfonamido)- ethanol	24448-09-7	NMePFOSAE	50000
Wellington	PFPeA	Perfluoropentanoic acid	2706-90-3	PFPeA	50000
Wellington	L-PFPeS	Perfluoropentanesulfonic acid	2706-91-4	PFPeS	46900
Wellington	6:2FTS	1H,1H,2H,2H perfluorooctanesulfonic acid	27619-97-2	6:2-FTS	47400
Wellington	N-EtFOSAA	N-ethylperfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	50000
Wellington	PFHxA	Perfluorohexanoic acid	307-24-4	PFHxA	50000
Wellington	MPFDoA	Perfluorododecanoic acid	307-55-1	PFDoDA	50000
Wellington	br-NMeFOSA	N-methylperfluoro-1-octanesulfonamide	31506-32-8	NMePFOSA	50000
Wellington	br-PFOA	Perfluorooctanoic acid	335-67-1	PFOA	50000
Wellington	PFDA	Perfluorodecanoic acid	335-76-2	PFDA	50000
Wellington	L-PFDS	Perfluorodecanesulfonic acid	335-77-3	PFDS	48200
Wellington	br-PFHxSK	Perfluorohexanesulfonic acid	355-46-4	PFHxS	45500
Wellington	FPrPA	3-Perfluoropropyl propanoic acid	356-02-5	3:3 FTCA	50000
Wellington	PFBA	Perfluorobutanoic acid	375-22-4	PFBA	50000
Wellington	L-PFBS	Perfluorobutanesulfonic acid	375-73-5	PFBS	44200
Wellington	PFHpA	Perfluoroheptanoic acid	375-85-9	PFHpA	50000
Wellington	L-PFHpS	Perfluoroheptanesulfonic acid	375-92-8	PFHpS	47600
Wellington	br-PFNA	Perfluorononanoic acid	375-95-1	PFNA	50000
Wellington	PFTeDA	Perfluorotetradecanoic acid	376-06-7	PFTeDA	50000
Wellington	PF4OFeA	Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	50000
Wellington	8:2FTS	1H,1H,2H,2H perfluorodecanesulfonic acid	39108-34-4	8:2-FTS	47900
Wellington	br-NEtFOSA	N-ethylperfluoro-1-octanesulfonamide	4151-50-2	NEtPFOSA	50000
Sigma Alrich	T6260-1G	Taurochenodeoxycholic Acid (TCDCA)	516-35-8	TCDCA	neat
Sigma Alrich	T0557-500MG	Taurodeoxycholic Acid (TDCA)	516-50-7	TDCA	neat
Wellington	L-PFNS	Perfluorononanesulfonic acid	68259-12-1	PFNS	48000
Wellington	PFTrDA	Perfluorotridecanoic acid	72629-94-8	PFTrDA	50000
Wellington	br-FOSA	Perfluorooctanesulfonamide	754-91-6	PFOSA	50000
Wellington	9Cl-PF3ONS	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	46600
Wellington	4:2FTS	1H,1H,2H,2H perfluorohexanesulfonic acid	757124-72-4	4:2-FTS	46700
Wellington	11Cl-PF3OUdS	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	47100
Wellington	L-PFDoS	Perfluorododecanesulfonic acid	79780-39-5	PFDoDS	48400
Wellington	FHpPA	3-Perfluoroheptylpropanoic acid	812-70-4	7:3 FTCA	50000
Wellington	PF5OHxA	Perfluoro(4-methoxybutanoic acid)	863090-89-5	PFMBA	50000
Wellington	FPePA	3-Perflurorpentylpropanoic acid	914637-49-3	5:3 FTCA	50000
Wellington	NaDONA	4,8-dioxa-3H-Perfluorononanoic acid	919005-14-4	DONA	47100
Wellington	M2PFOA	Perfluoro-n-(1,2-$^{13}C_2$)octanoic acid	STL00623	$^{13}C_2$-PFOA	50000
Wellington	MPFOA	Perfluoro-n-(1,2,3,4-$^{13}C_4$)octanoic acid	STL00990	$^{13}C_4$-PFOA	50000
Wellington	MPFOS	Perfluoro-1-(1,2,3,4-$^{13}C_4$)octanesulfonic Acid	STL00991	$^{13}C_4$-PFOS	47800
Wellington	MPFBA	Perfluoro-n-($^{13}C_4$)butanoic acid	STL00992	$^{13}C_4$-PFBA	50000
Wellington	MPFHxA	Perfluoro-n-(1,2-$^{13}C_2$)hexanoic acid	STL00993	$^{13}C_2$-PFHxA	50000
Wellington	MPFHxS	Perfluoro-1-hexane($^{18}O_2$)sulfonic acid	STL00994	$^{18}O_2$-PFHxS	47300
Wellington	MPFNA	Perfluoro-n-(1,2,3,4,5-$^{13}C_5$)nonanoic acid	STL00995	$^{13}C_5$-PFNA	50000
Wellington	MPFDA	Perfluoro-n-(1,2-$^{13}C_2$)decanoic acid	STL00996	$^{13}C_2$-PFDA	50000
Wellington	MPFUdA	Perfluoro-n-(1,2-$^{13}C_2$)undecanoic acid	STL00997	$^{13}C_2$-PFUnA	50000
Wellington	M8PFOA	Perfluoro-n-($^{13}C_8$)octanoic acid	STL01052	$^{13}C_8$-PFOA	50000
Wellington	M8PFOS	Perfluoro-1-($^{13}C_8$)octanesulfonic acid	STL01054	$^{13}C_8$-PFOS	47800
Wellington	M8FOSA	Perfluoro-1-($^{13}C_8$)octanesulfonamide	STL01056	$^{13}C_8$-FOSA	50000
Wellington	M4PFHpA	Perfluoro-n-(1,2,3,4-$^{13}C_4$)heptanoic acid	STL01892	$^{13}C_4$-PFHpA	50000
Wellington	M5PFPeA	Perfluoro-n-($^{13}C_5$)pentanoic acid	STL01893	$^{13}C_5$-PFBA	50000
Wellington	M2PFTeDA	Perfluoro-n-(1,2-$^{13}C_2$)tetradecanoic acid	STL02116	$^{13}C_2$-PFTeDA	50000
Wellington	d5-N-EtFOSAA	N-ethyl-d_5-perfluoro-1-octanesulfonamidoacetic acid	STL02117	d_5-NEtFOSAA	50000
Wellington	d3-N-MeFOSAA	N-methyl-d_3-perfluoro-1-octanesulfonamidoacetic acid	STL02118	d_3-NMeFOSAA	50000
Wellington	M3HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)($^{13}C_3$)propanoic acid	STL02255	$^{13}C_3$-HFPO-DA	50000
Wellington	d7-N-MeFOSE	2-(N-methyl-d_3-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02277	d_7-NMeFOSE	50000
Wellington	d9-N-EtFOSE	2-(N-ethyl-d_5-perfluoro-1-octanesulfonamido)ethan-d_4-ol	STL02278	d_9-NEtFOSE	50000
Wellington	M2-6:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)octanesulfonic Acid	STL02279	$^{13}C_2$-6:2FTS	47500
Wellington	M2-8:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)decanesulfonic Acid	STL02280	$^{13}C_2$-8:2FTS	47900
Wellington	M3PFBS	Perfluoro-1-(2,3,4-$^{13}C_3$)butanesulfonic acid	STL02337	$^{13}C_3$-PFBS	46500
Wellington	M2-4:2FTS	1H,1H,2H,2H-perfluoro(1,2-$^{13}C_2$)hexanesulfonic Acid	STL02395	$^{13}C_2$-4:2FTS	46700
Wellington	M5PFHxA	Perfluoro-n-(1,2,3,4,6-$^{13}C_5$)hexanoic acid	STL02577	$^{13}C_5$-PFHxA	50000
Wellington	M9PFNA	Perfluoro-n-($^{13}C_9$)nonanoic acid	STL02578	$^{13}C_9$-PFNA	50000
Wellington	M6PFDA	Perfluoro-n-(1,2,3,4,5,6-$^{13}C_6$)decanoic acid	STL02579	$^{13}C_6$-PFDA	50000
Wellington	M7PFUdA	Perfluoro-n-(1,2,3,4,5,6,7-$^{13}C_7$)undecanoic acid	STL02580	$^{13}C_7$-PFUnA	50000
Wellington	M3PFHxS	Perfluoro-1-(1,2,3-$^{13}C_3$)hexanesulfonic acid	STL02581	$^{13}C_3$-PFHxS	47300
Wellington	M3PFBA	Perfluoro-n-(2,3,4-$^{13}C_3$)butanoic acid	STL02680	$^{13}C_3$-PFBA	50000
Wellington	MPFDoA	Perfluoro-n-(1,2-$^{13}C_2$)dodecanoic acid	STL02703	$^{13}C_2$-PFDoDA	50000
Wellington	d-N-MeFOSA	N-ethyl-d_5-perfluoro-1-octanesulfonamide	STL02704	d_5-NEtFOSA	50000
Wellington	d-N-EtFOSA	N-methyl-d_3-perfluoro-1-octanesulfonamide	STL02705	d_3-NMeFOSA	50000
Wellington	M3PFPeA	Perfluoro-n-(3,4,5-$^{13}C_3$)pentanoic acid	STL03269	^{13}C3-PFPeA	50000

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1.0 SCOPE & APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of per- and polyfluoroalkyl substances (PFAS) in Table 1 in aqueous (all non-potable water and leachate) and solid (soil, biosolids, sediment) samples by liquid chromatography/mass spectrometry (LC-MS/MS). This SOP meets the requirements specified in ASTM D8421 and also EPA SW-846 8327.

The method calibrates and quantifies PFAS analytes using isotopically labeled standards. Where linear and branched isomers are present in the sample and either qualitative or quantitative standards containing branched and linear isomers are employed, the PFAS analyte is reported as a single analyte consisting of the sum of the linear and branched isomer concentrations.

The instrumental portion of this method is for use only by analysts experienced with LC-MS/MS or under the close supervision of such qualified persons. The laboratory must demonstrate the ability to generate acceptable results using the procedure in Sections 11.3.1 and 11.4.

By their very nature, many components of PFAS present analytical challenges unique to this class of analytes. For example, PFAS analytes readily adhere to the walls of the sample containers and may also stratify in the container.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes that can be determined by this SOP and the associated LOQ are provided in Table 1, Appendix A.

LOQs are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detections and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL and LOQ are always adjusted to account for actual amounts used and for dilution.

2.0 SUMMARY OF METHOD

A 5 mL aqueous sample is collected and processed in the same collection tube to limit analyte loss; extra samples must be collected for duplicates/triplicates and matrix spikes. Samples are shipped to the lab at a temperature between 0 °C and 6 °C and analyzed within 28 days of collection. All samples and associated QC samples are spiked with isotopically labeled compounds (aka surrogates or isotope dilution standards) and co-solvated with 5 mL of methanol before being shaken for 2 minutes. QC samples such as laboratory control and matrix spike samples are spiked with target PFAS in addition

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to the labeled surrogates. The samples are then filtered through a polypropylene filter. Acetic acid (~10 µL) is added to all samples to adjust to pH ~4 and analyzed by LC-MS/MS. Sample concentrations are determined by isotope dilution / extracted internal standard quantification using isotopically labeled compounds added to the samples before co-solvation and filtration.

If samples (such as sludge, pretreatment, or wastewater influent) contain more than ~1.0 g/L suspended or settled solids, sample pH is adjusted to pH ~9 (with ammonium hydroxide) before being shaken, filtered, and acidified as normal.

NOTE: Since contact with surfaces may bias data, a 5.0 mL sample is collected in a graduated 15 mL polypropylene tube in the field and the entire sample is processed in the lab. Once this 5.0 mL sample is spiked according to protocol and methanol is added, the sample is filtered into another 15 mL polypropylene tube without analyte loss. For accurate volume, the weight of the 15 mL polypropylene tube may be taken before and after sampling. The density of water is assumed to be 1.0 g/mL.

Individual PFAS analytes and surrogates are identified through peak analysis of the quantification and confirmation ions, where applicable. Specific ion transitions monitored for all target analytes and surrogates are listed in Table 2, along with example retention times.

Quantitative determination of target analyte concentrations is made with respect to an isotopically labeled PFAS standard; the concentrations are then used to convert raw peak areas in sample chromatograms to final concentrations. Results for target analytes are recovery corrected by the method of quantification (i.e., isotope dilution / extracted internal standard quantification). The final report lists the surrogate recoveries and the concentration of PFAS in ng/L, if detected, or as a non-detect at the LOQ, if not detected.

Solid samples are shipped to the lab at a temperature between 0 °C and 6 °C and analyzed within 28 days of collection. A sample (2 g) is transferred to a polypropylene tube, spiked with surrogates (all samples) and target PFAS compounds (laboratory control and matrix spike samples). The analytes are shaken for an hour with 10 mL of methanol:water (50:50) under basic condition (pH ~ 9-10, adjusted with ~20 µL ammonium hydroxide). The samples are centrifuged, and the extract is filtered through a polypropylene filter unit, leaving the solid behind. Acetic acid (~50 µL) is added to the filtered sample extracts to adjust to pH ~3-4 and then analyzed by LC/MS/MS.

3.0 INTERFERENCES

Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample containers and caps, and other sample processing equipment and lead to discrete artifacts and/or elevated baselines in the chromatograms. Method analytes may also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, autosampler vials, etc. Supplies and equipment are demonstrated to be free from interference (no analyte detected > ½ LoQ) by evaluation of routine method blanks (MB). If found, measures should be taken to remove the contamination or data should be qualified; subtracting blank values from sample results is not permitted.

Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample matrix.

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4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

- **Extracted Internal Standard (EIS) quantification** – The response of the target compound is compared to the response of the isotopically labeled analog of another compound with chemical and retention time similarities.
- **Isotope dilution (ID) quantitation** – A means of determining a naturally occurring (native) compound by reference to the same compound in which one or more atoms has been isotopically enriched. The labeled PFAS are spiked into each sample and allow identification and correction of the concentration of the native compounds in the analytical process.
- **Isotopically labeled compound** – An analog of a target analyte in the method which has been synthesized with one or more atoms in the structure replaced by a stable (non-radioactive) isotope of that atom. Common stable isotopes used are 13C (Carbon-13) or Deuterium (D or 2H). These labeled compounds do not occur in nature, so they can be used for isotope dilution quantitation or other method-specific purposes.
- **Multiple Reaction Monitoring (MRM)** – application of selected reaction monitoring to multiple product ions from one or more precursor ions.
- **Per- and Polyfluoroalkyl Substances (PFAS)** – synthetic organofluorine chemical compounds with multiple fluorine atoms that includes PFOA, PFOS, GenX, and many other chemicals. PFAS have a hydrophobic and oleophobic fluorinated “tail” and a hydrophilic “head” making them surfactants. The definition does not include mass-labeled surrogates or internal standards.
- **Precursor ion** – ion that reacts to form product ions or undergoes specified neutral losses.
- **Product ion** – ion formed as the product of a reaction involving a precursor ion.

5.0 HEALTH & SAFETY

Contact your supervisor or local safety coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

The following sections provide general health and safety information about chemicals and materials that may be present in the laboratory.

- The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
- PFOA has been described as likely to be carcinogenic to humans. Pure standards should be handled by trained personnel, with suitable protection to skin and eyes, and care should be taken not to breathe the vapors or ingest the materials.
- The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (EHS) policies and procedures specified in this SOP and in the Pace® Chemical Hygiene / Safety Manual (COR-MAN-0001)

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- Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.
- Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. For procedures that require use of acids, use acids in a fume hood whenever possible with PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. For procedures that that emit large volumes of solvents (evaporation/concentration processes), these activities must be performed in a fume hood or apparatus that reduces exposure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME & STORAGE

The laboratory provides containers for the collection of samples upon client request. Refer to *Sample Container Shipping* SOP [Admin SOP ENV-SOP-WCOL-0006] for procedures related to preparation of bottle kits for the test method(s) associated with this SOP.

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project. All sample containers must have linerless HDPE or polypropylene caps.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

The laboratory will provide containers for the collection of samples upon client request for analytical services.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

Container Type, Minimum Sample Amount, Preservation, and Holding Time Requirements:

Matrix	Container Size & Type	Required Sample Amount[1]	Preservation	Holding Time
Aqueous	3 x 15 mL polypropylene (PP)	5 mL	Thermal: 4 ± 2°C Chemical: None	Collection to Analysis: 28 days
Solid [Soil and sediment]	3 oz / 90 mL linerless PP straight sided	2 g	Thermal: 4 ± 2°C Chemical: None	Collection to Analysis: 28 days

[1] *Amount of sample required for each discrete test. For aqueous matrices, each sample container should contain 5 mL (± 0.5 mL)*

Thermal preservation is checked and recorded on receipt in accordance with *Sample Receiving* SOP [Admin SOP ENV-SOP-WCOL-0007]. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at 4 ± 2°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at 4 ± 2°C until sample analysis.

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After analysis, samples are retained as stated in the Pace® standard terms and conditions, unless otherwise specified in the analytical services contract. Samples are then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT & SUPPLIES

NOTE: Refer to the Major Operational Equipment List [QA Control Log ENV-FRM-WCOL-0213] for specific details regarding the equipment utilized during this procedure.

7.1 Equipment

- Analytical balance – Capable of weighing 0.0001 g
- Top loading balance – Capable of weighing 0.01 g
- Calibrated mechanical variable volume pipettes with disposable HDPE or polypropylene tips (10 µL to 10 mL) – used for preparation of calibration standards and spiked samples
- Point of Use water preparation system – Millipore Synergy – Used for QC samples to verify the systems are PFAS free; routinely tested to confirm absence of target analytes
- Analog or digital vortex mixer, single or multi-tube (Fisher Scientific 02-215-452, or equivalent)
- Volumetric flasks, Class A
- Variable speed mixing table (VWR Model 3500 Orbital Shaker, or equivalent)
- LC System – Agilent Model 1260, with Degasser (G4225A), Binary Pump (G1312B), Autosampler (A1329B), Thermostat (G1330B), Column Compartment (G1316A); Shimadzu Model LC-30AD, with Communication Bus Module (CBM-20A), Column Oven (CTO-30A), Degassing Unit (DGU-20A5R), Autosampler (SIL-20AC XR); or equivalent.

  Note: PFAS can build up in the PTFE solvent transfer lines and PTFE solvent frits. To prevent long delays in purging high levels of PFAS from the LC solvent lines, PEEK tubing and stainless-steel frits are used.
- Tandem Mass Spectrometer (MS/MS) – Sciex 4500 or 5500 MS/MS, in negative ion electrospray ionization (ESI) mode. A minimum of 10 scans across the chromatographic peak is required to ensure adequate precision.
- Analytical Column - Agilent Zorbax RRHD Eclipse Plus C18, 2.1 x 50 mm (1.8 µm) analytical column (Agilent Part # 959757-902), or equivalent
- Guard cartridge – SecurityGuard Cartridges: Gemini C18, 2-3 mm ID, 10/pk; Part# AJ0-7596/AJ0-7597; Phenomenex Part# KJ0-4282 (SecurityGuard Guard Cartridge Kit), or equivalent
- Trap/delay column – Agilent Zorbax Eclipse Plus C18, 2.1 mm, 1.8 um (Agilent Part# 821725-901), or equivalent

7.2 Supplies

- Polypropylene syringes, 20-mL, with luer lock, Fisher Scientific 14-817-176 or equivalent.

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- Syringe, 10 or 25-mL filter-adaptable glass, with luer lock, or equivalent. Reusable glass syringes must be cleaned before and after each use.
- Polypropylene syringe-driven filter units (0.22 µm), Restek Cat # 28935 or equivalent, demonstrated contaminant free below 1/2 MRL. Alternative: Phenomenex cat # AF8-6701-12
- 15- and 50-mL conical polypropylene tubes with polypropylene screw caps for preparing and storing extract solutions and collecting/preparing samples (VWR 10026-076 and Fisher Scientific 05-538-60, or equivalent)
- pH Paper, ranges 4-7 and 5-9, Supelco Cat # 1.09542.0001 and 1.09533.0001, or equivalent
- Reference matrix: Aqueous – reagent water
- Reference matrix: Solid – Ottawa sand (Fisher S23-3, or equivalent)
- Polypropylene vial, 11 mm, 250 µL and snap top (Agilent Part# 5190-3155 and 5182-0542, or equivalent)
- Extract/Standard storage containers – 15-mL, 8-mL, or 4-mL narrow-mouth HDPE container - Thermo Scientific item# 2002-9050, 2002-9025, 2002-9125; 2.0-mL screw-top polypropylene cryogenic vials – Grainger item# 6EMV1;1.5-mL snap-cap polypropylene microcentrifuge tubes - Fisher item# 05-408-129; or equivalent

8.0 REAGENTS & STANDARDS

NOTE: Other volumes of standards and reagents may be prepared to account for expected usage. As long as all ratios are kept constant, this is not considered a deviation from the approved procedure.

NOTE: All stored reagents and standards must be labeled as required by the Preparation and Documentation of Laboratory Standards and Reagents SOP [QA SOP ENV-SOP-WCOL-0083], the Contingency and Emergency Preparedness Plan [HS SOP ENV-SOP-WCOL-0015], the Safety Manual [Corp Manual COR-MAN-HSE], and the Laboratory Quality Manual [ENV-MAN-WCOL-0001].

NOTE: Reagents may be purchased from any approved vendor that can provide a certificate of analysis. All standards must be prepared using certified reference materials. Follow manufacturer expiration date unless stated otherwise below.

NOTE: Reagents prepared by the laboratory may be stored in either glass or HDPE containers. Proper cleaning procedures must be followed prior to using the containers.

8.1 Reagents

- Reagent Water – Unless otherwise indicated, references to water shall be understood to mean Type 1 reagent water. It shall be demonstrated that this water does not contain contaminants at concentrations sufficient to interfere with the analysis.
    - Mobile Phase A (95:5 Water:Acetonitrile with 10mM ammonium acetate) – to prepare 1 L, aliquot 2 mL of aqueous 5M ammonium acetate in 948 mL of water and 50 mL of acetonitrile. Alternatively, dissolve 0.77 g of ammonium acetate into 950 mL of water and 50 mL of acetonitrile. Reprepare and replace every 96 hours (4 days).

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- Acetic acid – ACS grade or equivalent, store at room temperature; Fisher cat# A35-500 or equivalent
- Acetonitrile – UPLC grade or equivalent, verified before use, stored at room temperature; Fisher A955-4 or equivalent.
    - Mobile Phase B: 100% Acetonitrile
- Ammonium acetate – LC/MS grade or equivalent, store under manufacturer specified conditions, replace 2 years after opening date; Fisher AM9071 or equivalent.
- Ammonium hydroxide – certified ACS+ grade or equivalent, 30% in water, store at room temperature; Fisher A669C-212 or equivalent
- Methanol – HPLC grade or better, 99.9% purity, store at room temperature; Fisher A452-4 or equivalent
- Isopropyl alcohol – HPLC grade or better, 99.9% purity, store at room temperature; Fisher A464-4 or equivalent
- Water – HPLC grade or better, 99.9% purity, store at room temperature; Fisher W6-4 or equivalent

8.2 Standards

Prepare standard solutions from materials of known purity and composition or purchase as solutions or mixtures with certification to their purity, concentration, and authenticity. Observe the safety precautions in Section 5.

Purchase of commercial standard solutions or mixtures is highly recommended for this method; however, when these are not available, preparation of stock solutions from neat materials may be necessary. If the chemical purity is 98% or greater, the weight may be used without correction to calculate the concentration of the standard.

When not being used, store standard solutions in the dark at 0-6 °C, unless the vendor recommends otherwise, in tightly sealed screw-capped vials. Place a mark on the vial at the level of the solution so that solvent loss by evaporation can be detected. Replace the solution if solvent loss has occurred.

The laboratory must maintain records of the certificates for all standards for traceability purposes. Copies of the certificates should be available to supplement the data packages and provide documentation that proper calculations were performed.

- **Surrogates/Isotope Dilution Standards (SUR)** - Isotopically labeled PFAS compounds; prepare the Surrogate 1 and Surrogate 2 solutions containing the isotopically labeled compounds listed below as standards in 95:5 methanol: water from stock standards. The list of isotopically labeled compounds listed in Table 2 represents the compounds that were available at the time this method was validated. Other isotopically labeled compounds may be used as they become available. Prepare the Surrogate 1 solution in a 15 mL centrifuge tube according to the table below:

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Standard / Reagent	Stock Conc (ng/mL)	Aliquot Volume (mL)	SUR 1 Conc (ng/mL)
MPFAC-24ES	1000	0.200	20
Water	Pure	0.500	-
Methanol	Pure	9.3	-

Prepare the Surrogate 2 solution in a 50 mL centrifuge tube according to the table below:

Standard / Reagent	Stock Conc (ng/mL)	Aliquot Volume (µL)	SUR 2 Conc (ng/mL)
d7-N-MeFOSE	50000	20	20
d9-N-EtFOSE	50000	20	20
D-N-EtFOSA	50000	20	20
D-N-MeFOSA	50000	20	20
MHFPO-DA	50000	20	20
Water	Pure	2500	-
Methanol	Pure	47400	-

- **Native Standards Solutions (NSS)** - Prepare a native standard spiking solution in 95:5 methanol: water, using stock standards containing the method target analytes listed below. This solution is used to prepare the calibration standards and to spike the known reference QC samples that are analyzed with every batch. Quantitative standards containing a mixture of branched and linear isomers should be used for method analytes if they are commercially available. Prepare the D8421 Intermediate and NSS according to the tables below. A 20X Spike Mix is then created from the original NSS solution, as detailed below.

Standard / Reagent	Stock Conc (ng/mL)	Aliquot Volume (µL)	D8421 Intermediate Conc (ng/mL)
PFPrA	50000	30	125
FOUEA	50000	30	125
FHUEA	50000	30	125
HQ-115	100000	15	125
Water	Pure	600	-
Methanol	Pure	11295	-

Standard/Reagent	Stock Conc (µg/L)	Aliquot volume (mL)	NSS Conc (µg/L)
1633 PFC Mix	2000	0.1	20
D8421-ID Intermediate	125	1.6	20
Acetic Acid	Pure	0.01	0.1%
Water	Pure	0.49	-
Methanol	Pure	7.8	-

Note: NSS analytical standard is made with a 10X dilution of a DM1633 analyte mix (Absolute Standards cat# 63735).

Standard/Reagent	Stock Conc (ng/L)	Aliquot volume (mL)	20X NSS Conc (ng/L)
D8421-ID NSS	20000	0.5	1000
Acetic Acid	Pure	0.01	0.1%

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Water	Pure	0.465	-
Methanol	Pure	9.025	-

- **ICV Standard Preparation** – Two separate solutions are prepared according to the tables below and used for Initial Calibration Verification purposes. MeFOSA is segregated into an independent solution (ICV B). All ICV compounds are prepared from a second source, separate from the source used for the original NSS, or a separate manufacturer lot if a true second source is not available. If a second source or manufacturer lot is unavailable, a separate preparation using the same stocks is acceptable. Prepare the final ICV analytical standards following the tables below.

Component	Aliquot for ICV A (µL)
D8421 ICV NSS	250
D8421-ID Surrogate 1	80
D8421-ID Surrogate 2	80
Water	4970
Acetic Acid	10
Methanol	4610
Final Volume	10000

Component	Aliquot for ICV B (µL)
MeFOSA 1000X	100
D8421-ID Surrogate 1	80
D8421-ID Surrogate 2	80
Water	4982
Acetic Acid	10
Methanol	4748
Final Volume	10000

Note: Immediately after preparation, shake samples for ~2 min. on a shaker table to fully homogenize.

- **Calibration Standard Solutions** - A series of calibration solutions containing the target analytes and the SUR is used to establish the initial calibration of the analytical instrument. The concentration of the method analytes in the solutions varies to encompass the working range of the instrument, while the concentrations of the SUR remain constant. The calibration solutions are prepared using methanol, water, acetic acid and the NSS and SUR solutions. The final solutions will match the solvent mix of prepared samples, which contain 50:50 water: Methanol with 0.1% acetic acid. Calibration standard solutions do not undergo filtration. Prepare the calibration standard solutions according to the table below:

Component[1]	L1	L2	L3	L4	L5	L6	L7	L8	L9	L10
20X NSS	50	100	200	500						
NSS					50	100	250	500	750	1000
SUR 1	80	80	80	80	80	80	80	80	80	80
SUR 2	80	80	80	80	80	80	80	80	80	80
Acetic acid	10	10	10	10	10	10	10	10	10	10
Water	4980	4977	4972	4957	4980	4977	4970	4957	4945	4932

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Methanol	4800	4753	4658	4373	4800	4658	4610	4373	4136	3898
Final Volume	10000	10000	10000	10000	10000	10000	10000	10000	10000	10000

[1]*All values listed are µL*

Concentrations for calibration solutions are presented in Table 3. All initial calibration requirements listed in Section 9.2 must be met.

Note: Immediately after preparation, shake samples for ~2 min. on a shaker table to fully homogenize.

Note: Additional calibration standards, at levels lower than the lowest calibration standard listed in the method, may be added to accommodate a lower limit of quantitation if the instrument sensitivity allows. Calibration standards at the high end of the calibration may be eliminated if the linearity of the instrument is exceeded or at the low end if those calibration standards do not meet the S/N ratio criterion of 3:1, so long as the required number of calibration points is met.

- **Qualitative Standards** - Standards that contain mixtures of the branched and linear isomers of the method analytes and that are used for comparison against suspected branched isomer peaks in field samples. These qualitative standards are not required for those analytes where the quantitative standards contain the branched and linear isomers. Qualitative standards that are currently commercially available include PFOA and PFNA. Create an intermediate standard containing all the qualitative standards at a concentration of 1 µg/mL (40uL of each stock into 2mL FV in MeOH) and use that to prepare the check standard according to the table below. Use the following Wellington Laboratories catalogue items to prepare the intermediate standard: T-PFOA, P355TMHxA (PFNA isomer), P4MOA (PFNA isomer), ipPFNA.

Component	Aliquot for Isomer Check STD (µL)
Isomer Intermediate STD	10
D8421 SUR 1	80
D8421 SUR 2	80
Water	4982
Acetic Acid	10
Methanol	4838
Final Volume	10000

Note: Immediately after preparation, shake samples for ~2 min. on a shaker table to fully homogenize.

- **Instrument blank** - An instrument blank is analyzed at the beginning of each analytical sequence, to demonstrate clean instrumental background, and after samples containing high levels of target compounds (e.g., calibration, CCV) to monitor carryover from the previous injection. The IBLK consists of clean reagent water and methanol fortified with the SURs. Prepare the IBLK using water and methanol, plus acetic acid and an appropriate volume of SUR. Prepare the IBLK from 5 mL water, 5 mL methanol, 10 µL acetic acid, and 80 µL each of SUR 1 and SUR 2. To aid in reproducibility, shake samples for ~2 min. on a shaker table to fully homogenize immediately after preparation.

9.0 PROCEDURE

9.1 Equipment Preparation

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9.1.1 Support Equipment

Incubators, water baths, refrigerator units, freezer units, bottle top dispensers, pipettes, thermometers, and ovens are maintained and verified as required by the Equipment and Instrumentation SOP [QA SOP ENV-SOP-WCOL-0080].

The balance is verified at the beginning of each analytical day using a certified weight set. Refer to the Equipment and Instrumentation SOP [QA SOP ENV-SOP-WCOL-0080] for balance verification procedures and acceptance criteria.

9.1.2 Instrument Set Up

All maintenance activities are listed in daily maintenance logs that are assigned to each instrument.

Mass Calibration – The mass spectrometer must undergo mass calibration to ensure accurate assignments by the instrument. This mass calibration must be performed at least annually to maintain instrument sensitivity and stability. Mass calibration must be repeated on an as-needed basis (e.g., QC failures, ion masses fall outside of the instrument required mass window, major instrument maintenance, or if the instrument is moved). Mass calibration must be performed using the calibration compounds and procedures prescribed by the manufacturer. The procedures used for mass calibration and mass calibration verification must evaluate an ion range that encompasses the ion range (Q1 and Q2 m/z) of the analytes of interest of this method.

Mass Calibration Verification – A mass calibration verification must be performed following mass calibration, prior to standard and sample analyses. Mass verification checks must also be performed after any subsequent mass calibrations. The laboratory must follow the instructions for the individual instrument software to confirm the mass calibration, mass resolution and peak relative response.

Multiple Reaction Monitoring (MRM) is required to achieve better sensitivity with the mass spectrometer than full-scan analysis. The ions to be monitored (quantitation and confirmation transitions, also referred to as quantitation and confirmation ions) for each native and SUR compound are given in Table 2.

The chromatographic conditions should be optimized for compound separation and sensitivity. The same optimized operating conditions must be used for the analysis of all standards, blanks, IDOCs, MDL and LCS standards, and samples. Different instruments may require slightly different operating conditions.

Retention Time (RT) calibration – After RT windows have been empirically confirmed for each analyte, the position of each method target analyte and SUR analyte peak shall be set once per ICAL using the midpoint standard of the ICAL curve.

Native target analyte and SUR analyte RTs must fall within 0.4 minutes (24 sec) of the predicted retention times from the midpoint standard of the ICAL. All branched isomer peaks identified in either the calibration standard or the qualitative standard must fall within the retention time window for that analyte.

For all target analytes with exact corresponding isotopically labeled analogs, target analyte peaks must elute within ±0.1 minutes (±6 sec) of the associated EIS.

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9.1.2.1 Routine Instrument Operating Conditions

Example Chromatographic Conditions

Step	Total Time (min)	Flow Rate (µL/min)	A: 5% Acetonitrile in Water w/ 10 mM Ammonium Acetate (%)	B: Acetonitrile (%)
0	0.00	400	98	2
1	0.01	400	98	2
2	0.20	400	80	20
3	4.00	400	60	40
4	7.00	400	45	55
5	9.00	400	25	75
6	9.50	400	5	95
7	10.50	400	5	95
8	10.55	400	1	99
9	12.55	400	1	99
10	13.00	400	98	2
11	14.00	400	98	2

Example Mass Spectrometer Conditions

Parameter	Setting or Value
Sample Loop	100 µL
Injection Volume	50 µL
Sampling Speed	15.0 µL/sec
Needle Stroke	52 mm
Column Oven Temperature	40°C
MRM Scan Window	120 sec
Curtain Gas (CUR)	30.0
Collision Gas (CAD)	9
Ion Spray Voltage (IS)	-4500.0 V
Temperature (TEM)	450°C
Ion Source Gas 1 (GS1)	40.0
Ion Source Gas 2 (GS2)	60.0

9.2 Calibration

9.2.1 Calibration Frequency

Prior to the analysis of samples, and after the mass calibration check has met all criteria in Section 9.1.2, each LC-MS/MS system must be calibrated at a minimum of 6 standard concentrations (Section 8.2 and Table 3).

This method procedure calibrates and quantifies 44 PFAS target analytes, using the isotopically labeled compounds added to the sample prior to extraction, by one of two approaches:

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• Isotope Dilution quantification (ID), whereby the response of the target compound is compared to the response of its isotopically labeled analog; twenty-four target compounds are quantified in this way.

• Extracted Internal Standard quantification (EIS), whereby the response of the target compound is compared to the response of the isotopically labeled analog of another compound with chemical and retention time similarities; twenty target compounds are quantified in this way.

Each LC-MS/MS system must be calibrated whenever the laboratory takes corrective action that might change or affect the initial calibration criteria, or if either the CCV or ISC acceptance criteria have not been met.

9.2.2 Calibration Levels

See Table 3 in the Appendix.

9.2.3 Calibration Sequence

A minimum of five contiguous calibration standards are required for a valid analysis when using a linear calibration model, with at least four of the five calibration standards being within the quantitation range (e.g., from the LOQ to the highest calibration standard). If a second-order calibration model is used, then a minimum of six calibration standards are required, with at least five of the six calibration standards within the quantitation range. The lowest level calibration standard must meet a signal-to-noise ratio of 3:1 and be at a concentration less than or equal to the Limit of Quantitation (LOQ). The calibration solutions contain the entire suite of SUR and target compounds. Calibration is verified at least once every ten field samples with a calibration verification (CCV), performed by analysis of a mid-level calibration solution.

Calibration standards must be analyzed in sequence from lowest to highest concentration to minimize the chance that carryover from a higher concentration standard will boost the area of a lower concentration standard. A typical sequence for days when calibration is required is shown below.

Description[1]	Comment
ICAL L1	
ICAL L2	
ICAL L3	
ICAL L4	
ICAL L5	
ICAL L6	
ICAL L7	
ICAL L8	Typically used as opening/bracket/closing CCV
ICAL L9	
ICAL L10	
IBLK	All targets must be <1/2 LOQ
ICV	%R must be 70 – 130%
Unknown Samples	Up to 10 Field Samples (not including MB, LCS, etc.)
CCV	%R must be 70-130%; begins and ends Analytical Sequence

[1]*See Table 3 for calibration concentrations*

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9.2.4 Calibration Evaluation

If the criteria for initial calibration are not met, inspect the system for problems and take corrective actions to achieve the criteria. This may require the preparation and analysis of fresh calibration standards. All initial calibration criteria must be met before any samples or required blanks are analyzed.

Instrument Sensitivity – Sufficient instrument sensitivity is established if a signal-to-noise ratio ≥ 3:1 can be achieved when analyzing the lowest concentration standard within the quantitation range that the laboratory includes in its assessment of calibration linearity (see Table 3).

If the ICAL evaluation criteria cannot be met, the analyst will have difficulty meeting ongoing QC criteria. It is recommended that corrective action is taken to reanalyze the ICAL standards, restrict the range of calibration, or select an alternate method of calibration.

If more than the minimum number of standards are analyzed and levels are excluded from the calibration, only the lowest or highest standards may be excluded, except as noted here. The removal of calibration levels from the interior of the curve is allowed only when there is sound technical reason for doing so and when the level is removed for all analytes; for example, when it can be proven that the wrong standard was analyzed for the calibration level or there is obvious evidence that the instrument malfunctioned during injection of the standard. The removal of any calibration level from the interior of the curve must be approved by the department supervisor/manager. Management approval and the rationale for the level removal must be documented and kept with the technical record.

Replacing a calibration standard may sometimes be needed to correct for a technical problem that occurred during analysis such as power failure, incomplete injection of the standard, or a similar situation. Replacement of one standard, when analyzed within 24 hours of original analysis time and replacing all analytes in the original standard, is permitted. The replacement of the standard must be approved by the department supervisor/manager; approval and the reason for replacement must be documented and kept with the technical record.

Once a passing calibration curve is generated, the analysis of samples may begin. Always end the sequence with a CCV.

9.2.4.1 Curve Fit

Either of two procedures are used to determine calibration function acceptability, depending on the calibration regression employed. Relative Standard Deviation (RSD, described below) evaluates the amount of deviation from the mean of a set of data. Relative Standard Error (RSE, described below) evaluates the difference between the true concentrations used to create the model and the measured concentrations. For calibration curves using average response factor regression, RSD is used to evaluate the curve fit; for curves using linear, weighted linear, or quadratic regressions, RSE is used to evaluate the curve fit.

Calculate the Relative Standard Deviation (RSD) of the Response Factor (RF) values of the initial calibration standards for each native compound and isotopically

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labeled compound using AVGRF regression using the equation below. The RSD must be ≤ 30% to establish instrument linearity.

$$\%\ \mathrm{RSD} = \frac{\mathrm{SD}}{\mathrm{AVG}} \times 100$$

Where:

SD = the standard deviation of the curve

AVG = the average response factor for the curve (y = AVG*x)

9.2.4.2 Relative Error

Calculation of RSE (expressed as %) is shown below. The RSE acceptance limit criterion for the calibration model is the same as the RSD limit (≤ 30 %).

$$RSE = 100 * \sqrt{\sum_{i=1}^{n} \left(\frac{x'_i - x_i}{x_i} \right)^2 / (n - p)}$$

Where:

xi = true amount of analyte in calibration level i, in mass or concentration units,

x'i = measured amount of analyte in calibration level i, in mass or concentration units,

p = number of terms in the fitting equation (average = 1, linear = 2, quadratic =3, cubic =4),

n = number of calibration points

9.2.4.3 Initial Calibration Verification

As part of the IDOC, each time a new Target Spike (NSS) is prepared, and once after each ICAL, analyze an ICV sample prepared from a second source (different from the source of the ICAL standards). If a second vendor is not available, then a different lot of the standard from the same vendor should be used. The ICV should be prepared and analyzed just like a CCV. Acceptance criteria for the ICV are identical to the CCVs: the calculated concentration for each analyte must be within ± 30% of the expected value. If measured analyte concentrations are not of acceptable accuracy, correct the problem and rerun the ICV. If the problem persists, repeat the ICAL. Samples are not to be analyzed until the ICAL has been verified by acceptable ICV accuracy.

9.2.4.4 Continuing Calibration Verification

After a passing MS resolution and a successful initial calibration is achieved and prior to the analysis of any samples, the calibration is verified by injecting an aliquot

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of the appropriate concentration ICAL standard, analyzed with the same conditions used during the ICAL. CCV is performed at the beginning of each analytical sequence (that does not directly follow an ICAL event), after every ten samples, and at the end of the analytical sequence. In this context, a "sample" is defined as a field sample. MBs, CCVs, LCSs, MSs, FDs, TBs and MSDs are not counted as samples. All CCV analyses are performed using the mid-level ICAL standard.

Calculate concentration for each native compound in the CCV using the equation in Section 10.3; calculate the recovery of each native and SUR compound by comparison to the expected concentration. The recovery of native and SUR compounds for the CCVs must be within 70 – 130% of expected concentrations. If the CCV criteria are not met, corrective action(s) and/or re-calibration are performed, with all affected samples in the batch either re-analyzed or qualified. Alternately, the analyst may immediately analyze two CCVs for confirmation. If both confirmation CCV analyses meet the recovery criteria, analysis may proceed; however, the analyst must rerun any samples that were analyzed after the failing CCV and before the 2 passing CCVs. If either of the 2 confirmation CCV analyses fails to meet the acceptance criteria, recalibrate the LC-MS/MS according to Section 9.2.

If an individual target compound recovery in a CCV is above the upper control limit and all associated samples are ND for that compound, the data for those samples may be reported. In such cases, a narrative statement must be included in the report indicating the specific compound result that was biased high in the CCV, and that samples were ND for that compound and thus reportable.

9.3 Sample Preparation

This test method is based upon a 5 mL (or 2 g) sample size per analysis. Analyze within 28 days of collection. Each batch of samples (20 or less) must contain a method blank, a reagent blank, a laboratory control sample, and an MRL check. Each batch of samples should also have a matrix spike and a duplicate or matrix spike duplicate. Depending on project objectives and/or plans, samples may be analyzed in duplicate.

Do not use any fluoropolymer articles or task wipes in these preparation procedures. Use only HDPE or polypropylene wash bottles and centrifuge tubes. Reagents and solvents for cleaning syringes may be kept in glass containers.

9.3.1 Homogenization & Subsampling

Subsampling is not allowed in routine application of this method. If a sample container is received with too much volume to be co-solvated in the same container (example: 10 mL of sample collected in 15 mL tube), perform a quantitative transfer to an appropriately sized vessel as described below.

If the sample is transferred to another container prior to diluting 1:1 with methanol, the original container must be solvent rinsed and the rinsate included in the solvent dilution of the sample. Shake by hand or vortex the rinse solvent in the original sample container for ~2 min to ensure quantitative transfer. The total volume of methanol added to the sample, including the volume used for quantitative transfer, should be equivalent to the sample

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volume. SUR must be spiked before the sample is transferred from the original sample container.

9.3.2 Aqueous Sample Processing

The entire 5.0 mL sample is used without transferring to another sample container. For greater volume accuracy, weigh the 15 mL polypropylene tube before and after sampling, or weigh the tube with sample, and then weigh the emptied and dried tube after analysis is complete. Allow the samples to warm to room temperature to determine the sample volume by mass difference. Assume the density of water to be 1.0 g/mL. The entire sample must be processed in the original container, otherwise results may be biased low. The entire collected sample is appropriately spiked.

All samples and QC are spiked with 40 µL each of the surrogate spiking solutions (20 µg/L in 95 % MeOH: 5 % water) for a concentration of 160 ng/L of the surrogates in the sample. All QC samples are then spiked with native standards: 40 µL of NSS for LCS/MS/MSD and 50 µL of 20X-NSS for MRLCS. To all samples, 5 mL of methanol is added, and the solution is mixed by shaker table for ~2 minutes (see below for additional steps for sludge samples).

After mixing, pH of sludge samples is adjusted to pH ~9 with ammonium hydroxide (~20 µL) and mixed again for ~2 minute. This step is not required unless high percent solids (≥0.1 %) are visually present or low recoveries were observed historically.

Prepared samples are filtered using a lock tip syringe to remove particulates in the samples. Before use, refer to syringe and filter cleaning protocols listed below. Centrifugation may aid in removal of particulates. After filtration, acetic acid is added (~10 µL for water samples and ~50 µL for ammonium hydroxide-prepared samples) to all samples to adjust the pH to ~4. An aliquot of the prepared sample is transferred to an LC vial and capped for analysis.

The final volume of the solution is calculated using the volume of the sample and 5.0 mL volume of the methanol. For quantitation purposes, since 5 mL of methanol was added to 5 mL of water, the combined volume is assumed to be 10.0 mL . If a sample is received with substantially more or less than 5 mL, the lab will adjust the volume of methanol used to co-solvate the sample to match the sample volume. If a sample is received with <4.5 or >6.5 mL, based on the gradations marked on the sample container, the lab will adjust the volume of methanol added to match, approximating to the nearest 0.5 mL increment. Example: sample is received with ~6.5 mL, so the lab adds 6.5 mL of methanol for co-solvation before filtration and analysis.

9.3.3 Syringe and Filter Cleaning

Either polypropylene or reusable glass luer lock syringes can be used. Polypropylene syringes are expected to be single use consumable items. If reusable glass syringes are used, then the syringe shall be cleaned between each use. A suggested method for cleaning the syringe between filtrations is to first use DI water in a wash bottle to rinse both the plunger and the syringe (at least 2 syringe volumes), then repeat with MeOH (at least 2 syringe volumes); the clean pieces should be laid out to dry on clean paper towels in a hood until needed for sample processing.

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Prior to use, rinse the filters with two 10-mL volumes of acetonitrile followed by two 10-mL volumes of methanol to ensure removal of PFAS contamination.

Note: Avoid attaching the filter to the syringe before removing the plunger from the syringe barrel, as the syringe disk filters can be compromised by the sudden vacuum created when the plunger is removed.

9.3.4 Solid Sample Processing

Mix the sample in its original jar. If it is impractical to mix the sample within its original container, transfer the sample to a larger container. Mix the sample thoroughly, stirring from the bottom to the top and in a circular motion along the sides of the jar, breaking particles up by pressing against the side of the container. The homogenized sample should be even in color and have no separate layers. Store the homogenized material in its original container.

Weigh out 2±0.2 g of homogenized sample or reference matrix, recorded to the nearest 0.01g, and place in a 15-mL polypropylene centrifuge tube. Every QC and field sample is then spiked with 40 µL each of the surrogate spiking solutions (20 µg/L in 95 % MeOH: 5 % water) for a concentration of 20 ng/g of the surrogates in the sample. QC samples are then spiked with native standards: 40 µL of NSS for LCS/MS/MSD and 50 µL of 20X-NSS for MRLCS.

To all samples, 5 mL of water and then 5 mL of methanol is added and hand shaken/vortexed for ~1 min. After vortexing, pH of the samples is adjusted to pH ~9-10 with ammonium hydroxide (~20 µL) and hand shaken/vortexed again for ~1 min. The sample tubes are placed on a shaker table for 1 h, then centrifuged at 1900 rpm for 10 min. The supernatant of the sample is filtered through a syringe driven filter unit to remove particulates in the samples and leave solids behind. Before use, refer to Syringe and Filter Cleaning protocols above. Acetic acid (~75 µL) is added to all samples to adjust to pH ~3-4 after filtration. An aliquot of the solution is transferred to an LC vial and capped, ready for analysis. The final volume of the solution is 10 mL for quantitation purposes since 10 mL of MeOH/water was added.

9.4 Analysis

Analysis of sample extracts for PFAS by LC-MS/MS is performed running manufacturer's data acquisition software. The mass spectrometer is run with unit mass resolution in the multiple reaction monitoring (MRM) mode.

Perform mass calibration, establish the operating conditions (Section 9.1.2), and perform an initial calibration (Section 9.2) prior to analyzing samples.

Only after all performance criteria have been met may blanks, MDLs, IDOC/LCSs, and samples be analyzed.

9.4.1 Preparation

Column Flush – Each day of analysis, the column must be thoroughly flushed with 100% Acetonitrile for at least 30 minutes to clear any accumulated impurities and interferences from the sample pathway and equilibrate the system. It is also good practice to open the purge valve(s) on the pumps for the first ~1 minute of flush time and/or use the “purge”

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function available as part of the pump capabilities (Shimadzu LC). Additionally, it is good practice to purge the autosampler injector flow path daily prior to analysis by using the "purge" function, if available. The column should then be equilibrated to the analysis starting conditions by flushing for approximately 15 mins with 50:50 Mobile Phase A:Mobile Phase B and finally approximately 15 mins with 98:2 Mobile Phase A:Mobile Phase B. Ensure that system pressure is stable.

Analytical Sequence – Following the daily column flush, two to three (2-3) high ICAL standard (L8, L9 or L10) injections and one blank injection will be made in order to prime the system before analyzing opening QC and client samples. Following these opening injections, an opening CCV (L8), an instrument blank, and an isomer check injection will be analyzed to demonstrate system suitability and the absence of system contamination.

9.4.2 Example Analytical Sequence

After a successful initial calibration has been completed, an analytical sequence for a batch of samples may be analyzed. An example analytical sequence is detailed below. The volume injected for samples and QCs must be identical to the volume used for calibration (Section 9.2). Standards and prepared samples must be brought to room temperature and vortexed prior to aliquoting into an instrument vial to ensure homogeneity of the solution.

- Calibration Verification Standard (CCV)
- Instrument Blank (IBLK)
- Isomer Check
- Method Blank / Reagent Blank
- MRL Check Sample
- LCS
- Samples (10 or fewer)
- CCV
- IBLK
- Samples (10 or fewer)
- CCV

If the results are acceptable, the closing CCV analysis may be used as the opening CCV analysis for the next analytical sequence.

If the response exceeds the calibration range for any sample, extracts are diluted as per Section 9.4.3 to bring all target responses within the calibration range.

Note: If the analytes that exceed the calibration range in the original analysis are known to not be of concern for the specific project (e.g., are not listed in a discharge permit), then the laboratory may consult with the client regarding the possibility of reporting that sample from the undiluted analysis.

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9.4.3 Sample Dilutions

If the measured concentration for any compound exceeds the calibration range of the system, dilute a subsample of the prepared sample with a prepared solution of 50:50 MeOH: Water with 0.1% acetic acid and analyze the dilution. If the compound still exceeds the calibration range, prepare a further dilution of the prepared sample and reanalyze. If the dilution of the prepared sample is less than or equal to 20X, the dilution solution will not be refortified with SUR. If the dilution of the prepared sample is greater than 20X, the dilution solution will be refortified with SUR.

10.0 DATA ANALYSIS & CALCULATIONS

10.1 Qualitative Identification

A native or SUR compound is positively identified in a standard, blank, sample, or QC sample when all criteria in Sections 10.1.1 through 10.1.3 are met.

10.1.1 Signal-to-noise Ratio (S/N)

Peak responses must be at least three times the background noise level (S/N 3:1). If the S/N ratio is not met due to high background noise, the laboratory should correct the issue if possible (e.g., perform instrument troubleshooting). If the S/N ratio is not met but the background is low, then the analyte is to be considered a non-detect.

10.1.2 RT Criteria

The retention time window of an unknown shall be within ±0.2 mins of the retention time of the analyte in a mid-level calibration standard. If this is not the case, re-analyze the sample to confirm if there was a shift in retention time during the analysis relative to a mid-level calibration standard. If the target analyte retention time has shifted and the associated EIS has not, refer to the analyte as not detected. If both the target analyte and the associated EIS shift outside of the +/- 0.2 min window, then report the target analyte as detected and issue a non-conformance memo that states that the analyte and EIS shifted outside of +/- 0.2 min window, presumably due to matrix interference. The retention time window used must be of sufficient width to detect earlier-eluting branched isomers. For all method analytes with exact corresponding isotopically labeled analogs, method analytes should elute within ± 0.1 minutes of the associated labeled analog compound.

10.1.3 Branched Isomer Integration

For concentrations at or above the method LOQ, the total (branched and linear isomer) quantification ion response to the total (branched and linear isomer) confirmation ion response ratio (ion ratio) shall fall within ± 30% of the average ratio observed in the initial calibration standards. The quantification and confirmation transitions may be switched to achieve the best sensitivity and to minimize MRM channels with interference. The transitions used need to be documented in the Level 4 data package accompanying the data.

The response of all isomers in the quantitative standards should be used to define ion ratios. In samples, the total response should include the branched isomer peaks that have been identified in either the quantitative or qualitative standard; the entire isomeric group

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shall be quantitated. If standards (either quantitative or qualitative) are not available for purchase, only the linear isomer can be identified and quantitated in samples. The ion ratio requirement does not apply for PFBA, PFPeA, PFOSA, NMeFOSE, NEtFOSE, PFPrA, PFMPA, PFMBA, FHUEA, or FOUEA because suitable secondary transitions are unavailable (not detectable or inadequate S/N). If there are peaks in an isomeric mixture that do not match the retention times of the standard, they may not be included in the integration.

The confirmatory ion ratios in "weathered samples" may not match the ion ratios in the calibration standards for the target analytes that may contain isomeric mixtures. The differences for PFHxS and PFOS were found in groundwater samples and may either be caused by different compositions used, weathering or degradation, or the affinity of the branched isomers to be more water soluble than the linear isomers, thus leaching into water from soil at a higher rate.

10.1.4 Manual Integration

Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument maintenance and/or method set-up. To assure that all manual integrations are justified and proper all manual integrations must be performed, documented, reviewed, and approved in accordance with corporate SOP ENV-SOP-CORQ-0006, Manual Integration. Refer to this SOP for guidance on manual integration techniques and required procedures.

10.2 Quantitative Identification

Concentrations of the target analytes are determined with respect to the Surrogate compounds (SUR, also called isotope dilution standards or extracted internal standards) which are added to the sample prior to extraction, using the response ratios or response factors from the most recent multi-level initial calibration (Section 9.2). Quantitation standard (SUR) associations are shown in Table 2. Other equations may be used if the laboratory demonstrates that those equations produce the same numerical result as produced by the equations below.

If there is no confirmatory transition for the analyte (refer to Table 2), and the presence of the analyte in the sample can't be confirmed with the primary transition and retention time, the analyte is not detected.

All results for aqueous samples will be reported in ng/L. All results for solid samples will be reported in ng/g, on a dry-weight basis, and the percent solids for each sample will be reported separately. All QC data will be reported with the sample results.

Unless specified otherwise by a regulatory authority or in a discharge permit, results for analytes that meet the identification criteria are reported down to the concentration of the LOQ (Section 11.3.1) established by the laboratory through calibration of the instrument.

Results for any analyte found in a sample or extract that has been diluted will be reported at the least dilute level for which the measured concentration is within the calibration range (e.g., above the LOQ for the analyte and below the highest calibration standard) and in which SUR compound recoveries are within their respective QC acceptance criteria. This may require reporting results for some analytes from different analyses.

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Recoveries of all associated SUR compounds will be reported for all field samples and QC standards.

10.3 Calculations

See the Laboratory Quality Assurance Manual for equations for common calculations.

Calculation	Formula	Variables
Relative Standard Error (RSE)	$RSE = 100 * \sqrt{\sum_{i=1}^{n} \frac{x'_i - x_i}{x_i}^2 / (n-p)}$	x_i = true amount of analyte in calibration level i, in mass or concentration units x'_i = measured amount of analyte in calibration level i, in mass or concentration units p = number of terms in the fitting equation (average = 1, linear = 2, quadratic =3, cubic =4), n = number of calibration points
Relative Standard Difference	$\%RSD = \frac{SD}{AVG} \times 100$	SD = Standard Deviation AVG = Average
Analyte Concentration [ng/L or ng/g]	$Concentration\ (ng/L\ or\ ng/g) = \frac{Area_N M_{SUR}}{Area_{SUR}(RF)} x \frac{1}{W_S}$	$Area_N$ = The measured area of the Q1 m/z for the native (unlabeled) PFAS $Area_{Sur}$ = The measured area at the Q1 m/z for the EIS (SUR). M_{SUR} = The concentration of the EIS (SUR) added (ng) RF = Average response ratio used to quantify target compounds by the isotope dilution / extracted internal standard method W_S = Sample volume (L) or dry weight (g)

11.0 QUALITY CONTROL & METHOD PERFORMANCE

11.1 Quality Control

Prepare the following QC samples with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action(s).

QC Check	Acronym	Frequency

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Method Blank	MB	1 per batch of 20 or fewer samples.
Reagent Blank	MB2	1 per batch of 20 or fewer samples.
Laboratory Control Sample	LCS	1 per batch of 20 or fewer samples.
Method Reporting Limit Check Sample	MRLCS	1 per batch of 20 or fewer samples.
Matrix Spike	MS	As requested
Matrix Spike Duplicate	MSD	As requested
Sample Duplicate	SD	1 per batch of 20 or fewer samples.
Surrogate (Isotopically-labeled quantitation standards)	SUR	Spiked into every field and QC sample

The minimum quality control requirements of this method consist of an initial demonstration of laboratory capability, analysis of samples spiked with isotopically labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

If the method is to be applied to a sample matrix other than water (e.g., soils, biosolids, sediments), the appropriate alternative reference matrix (Section 7.2) is substituted for the reagent water matrix in all performance tests.

The laboratory must make an initial demonstration of the ability to generate acceptable precision and recovery with this method. This demonstration is described in Section 11.4.

Analyses of method blanks (MBs) are required on an on-going basis to demonstrate the extent of background contamination in any reagents or equipment used to prepare and analyze field samples. The procedures and criteria for analyses of a MBs are described in Section 11.1.1.

The laboratory must spike all samples with isotopically labeled compounds to monitor method performance. This procedure is described in Section 11.3. When results of these spikes indicate atypical method performance for samples, the samples may be diluted to evaluate whether the performance issue is caused by the sample matrix. Procedures for dilution are given in Section 9.4.2.

The laboratory must, on an ongoing basis, demonstrate that the analytical system is in control through calibration verification and the analysis of ongoing precision and recovery standards, spiked at low (MRLCS) and mid-level concentrations (LCS), and blanks. These procedures are given in Sections 11.1.2 and 11.1.1.

11.1.1 Method Blanks (MB)

A method blank is analyzed with each extraction batch to demonstrate freedom from contamination. The matrix for the MB must be similar to the sample matrix for the batch (e.g., reagent water blank or Ottawa sand blank [Section 7.2]).

Analyze the prepared method blank aliquot before the analysis of the LCSs.

If any PFAS is found in the MB 1) at a concentration greater than ½ the LOQ for the analyte or 2) at a concentration greater than one-tenth the concentration in a sample in the extraction batch, whichever is greater, analysis of samples must be halted, and the problem corrected. Other project-specific requirements may apply; therefore, the laboratory may

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adopt more or less stringent acceptance limits for the method blank at their discretion. If the contamination is traceable to the extraction batch, samples affected by the blank must be re-prepared and analyzed, provided enough sample volume is available and the samples are still within holding time. Reanalysis or re-extraction is not required if the samples are not impacted. Samples are not impacted when the MB detection is not present in the sample, or the sample concentration is ≥10x the concentration of the detection in the MB.

If continued re-testing results in repeated blank contamination, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority.

11.1.2 Laboratory Control Sample (LCS)

Analyze the prepared LCS and MRLCS to ensure the analytical process is under control.

Compute the percent recovery of the native compounds by the appropriate quantification method depending on the compound. Compute the percent recovery of each isotopically labeled and native compound using the equation below:

$$Recovery\ \% = \frac{Concentration\ Found\ (ng/L)}{Concentration\ Spiked\ (ng/L)} \times 100$$

For the native compounds and isotopically labeled compounds, compare the recovery to the LCS limits. Analyte recoveries must be within in-house limits if project limits are not provided; otherwise, project limits must be met. Preliminary in-house acceptance criteria of 70-130% recovery must be used for aqueous LCS analyses until method limits based on an interlaboratory study are published. The acceptance range for MRLCS analyses is 50-150% recovery.

For solid matrix LCS, all recovery limits for target compounds are 50-150%.

If all compounds meet the acceptance criteria, system performance is acceptable, and analysis of samples may proceed. If, however, any individual concentration falls outside of the given range, the preparation processes are not being performed properly for that compound. In this event, correct the problem, re-prepare the extraction batch and repeat the ongoing precision and recovery test.

11.1.3 Matrix Spike (MS)

Analysis of an MS may be required in each extraction batch. Assessment of method precision can be accomplished by preparation and analysis of a matrix spike duplicate (MSD).

Typically, a minimum of one pair of sample bottles is spiked as MS/MSD for every 20 samples analyzed. MS/MSD samples are spiked in the same manner as the mid-level LCS.

Analyte recoveries may exhibit matrix effect. For matrix spike samples, acceptance criteria for recovery should match LCS limits. If the % recovery falls outside of the acceptable range, corrective action must occur. The initial corrective action will be to check all calculations. If the calculations are correct, check the recovery of that analyte in the LCS.

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If the recovery of the analyte in the LCS is within limits, then matrix interference has been demonstrated and the laboratory operation may proceed. Analytical reports will show qualifier flags in such cases.

If the recovery for any analyte is outside the acceptance criteria for the matrix spike and the LCS, the laboratory is out of control and corrective action will be taken. Corrective action may include repreparation and reanalysis of the batch. A narrative statement will be added to document the corrective action taken.

RPDs for MS/MSDs should be ≤30%. If the RPD falls outside of the acceptable range, corrective action must occur. The initial corrective action will be to check all calculations. If the calculations are correct, check the recovery of that analyte in the LCS. If the recovery of the analyte in the LCS is within limits, then matrix interference has been demonstrated and the laboratory operation may proceed. Analytical reports will show qualifier flags in such cases.

11.2 Instrument QC

Perform the following checks to verify instrument performance. Refer to Appendix B for acceptance criteria and required corrective action.

Instrument Check	Acronym	Frequency
Initial Calibration	ICAL	Prior to analysis and on as needed basis
Initial Calibration Verification	ICV	Following each ICAL
Initial Calibration Blank	ICB	Immediately following highest concentration ICAL standard and daily prior to analysis
Continuing Calibration Verification	CCV	At the beginning and every 10 field samples
Continuing Calibration Blank	CCB	After each CCV
Retention Time Windows		After ICAL

11.2.1 Instrument Blank (IBLK)

One instrument blank (IBLK) is analyzed immediately following the highest ICAL standard analyzed, each analysis day prior to sample analysis, and following each bracketing CCV in a sequence, to check for carryover and instrument contamination. The concentration of each analyte must be ≤ 1/2 the LOQ. If the IBLK does not pass this requirement after the highest ICAL standard, the calibration must be performed using a lower concentration for the highest standard until the acceptance criteria is met.

11.2.2 Qualitative Isomer Check

A qualitative identification standard (Section 8.2) containing all available isomers (branched and linear) is analyzed once during the calibration sequence, to confirm the retention time of each linear and known branched isomer or isomer group. All required branched isomer peaks must be present and visibly resolved from their corresponding linear peak.

11.3 Method Performance

Routine method performance is validated through analysis of matrix-specific reference samples, including spikes and PTs. Ongoing method performance is monitored through QC samples

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analyzed alongside samples. The parameters monitored include percent recovery of SUR compounds, blank concentrations, and native compound recoveries.

The specifications contained in this method can be met if the apparatus used is calibrated and maintained properly. The standards used for initial calibration, calibration verification, and for initial (Section 11.4) and ongoing precision and recovery may be prepared from the same source; however, the use of a secondary source for calibration verification is highly recommended whenever available. If standards from a different vendor are not available, a different lot number from the same vendor can be considered a secondary source. An LC-MS/MS instrument will provide the most reproducible results if dedicated to the settings and conditions required for determination of PFAS by this method.

To assess method performance on the sample matrix, the laboratory must spike all samples with the SUR solution (Section 8.2). Analyze each sample according to the procedures in this SOP. Compute the percent recovery of the SUR compounds using the equation in Section 11.1.2. The recovery of each SUR compound must be assessed and be within the control limits; the lab must maintain records of these assessments. If the recovery of any compound falls outside of these limits, method performance is unacceptable for that compound in that sample. If the recovery cannot be brought within the normal range, aqueous samples are diluted and smaller amounts of solid matrices are prepared and analyzed, per Section 9.4.2.

SUR preliminary in-house acceptance criteria of 70-130% must be used until method acceptance values are set based on the ILS.

11.3.1 Method Validation

Refer to corporate SOP ENV-SOP-CORQ-0011 for general requirements and procedures for method validation.

Establish detection limits (DL) and limits of quantitation (LOQ) at initial method set up and verify the DL and LOQ on an on-going basis thereafter. Refer to corporate policy and/or SOP for DL and LOQ requirements and procedures.

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to the *Demonstration of Capability* SOP [ENV-SOP-WCOL-0077] for more information.

IDOC - To establish the ability to generate acceptable precision and recovery, the laboratory must perform the following operations for each sample matrix type to which the method will be applied by that laboratory.

Prepare and analyze four aliquots of the matrix type to be tested, prepared at one to four times the LOQ. At least one method blank, matching the matrix being analyzed, must be prepared with the IDOC batch. If more than one MB was prepared and analyzed with the IDOC batch, all

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blank results must be reported. All sample processing steps that are to be used for processing samples must be included in this test.

Using results of the set of four analyses, compute the average percent recovery (R) of the extracts and the relative standard deviation (RSD) of the concentration for each target and SUR compound.

For each native and isotopically labeled compound, compare RSD and R with the corresponding limits for initial precision and recovery. If RSD and R for all compounds meet the acceptance criteria, system performance is acceptable, and analysis of blanks and samples may begin. If, however, any individual RSD exceeds the precision limit or any individual R falls outside the range for recovery, system performance is unacceptable for that compound. Correct the problem and repeat the test.

12.0 DATA REVIEW & CORRECTIVE ACTION

12.1 Data Review

The data review process of Pace® Analytical Services includes a series of checks performed at different stages of the process by different people to ensure that SOPs were followed, the analytical record is complete, and properly documented, QC criteria were met, proper corrective actions were taken for QC failure and other nonconformance(s), and test results are reported with proper qualification, when necessary.

The review and checks that are performed by the employee performing the task is called primary review.

All data and test results are also peer reviewed.

This process, known as secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented, and approved in accordance with the Pace® Analytical Services SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

Lastly, a third-level review, called a completeness check, is performed by reporting or project management staff to verify the test report is complete.

Refer to *Data Review* SOP [QA SOP ENV-SOP-WCOL-0075] for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is required when QC or sample results are not within acceptance criteria.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking

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corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 POLLUTION PREVENTION & WASTE MANAGEMENT

Pace® proactively seeks ways to minimize waste generated during work processes. Some examples of pollution prevention include but are not limited to reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practices comply with all applicable federal and state laws and regulations. Excess reagents, samples, and method process wastes are characterized and disposed of in an acceptable manner in accordance with the Pace® Chemical Hygiene Plan / Safety Manual. Refer to this manual for these procedures.

14.0 MODIFICATIONS

The procedures in this SOP have been modified from the reference test method as follows:

Modification	Test Method Procedure	Justification for Modification
Isotope Dilution / Extracted Internal Standard Quantitation	External Standard Quantitation	Automatic recovery correction for target analytes
Mobile Phase B updated to 100% Acetonitrile	Mobile Phase B is 95:5 Acetonitrile: Water, with 10mM ammonium acetate	Method simplification; match DM1633 for better interoperability in the lab
Changes to Gradient Curve	See Section 9.1.2.1	Shorten the run time (21 min -> 14 min), saving time and reagents
Change Delay Column to Waters C18-AX	“Reverse Phase C18 column”	Better performance; Change suggested by ILS coordinator
Change Analytical Column to Agilent C18	UHPLC CSH Phenyl-Hexyl, 2.1x100mm, 1.7 µm	Match DM1633 and ID method for better interoperability in the lab
Added Guard Cartridge: Phenomenex Gemini C18	Not Used	Extend analytical column lifetime
Changed Flow Rate to 0.4 mL/min for whole run	Flow rate of 0.3 mL/min for the first 13 min, then 0.4 mL/min from 14-21 min	Shorten the run time (21 min -> 14 min), saving time and reagents; consistent flow rate across entire run

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Using separate additions of water and methanol during solids sample preparation	Add 10 mL of a premixed solution of 1:1 water:methanol	Premixed water:methanol solutions immediately begin to evaporate and do not maintain 1:1 ratio, which affects surrogate recovery.
Using separate additions of water, methanol, and acetic acid for IBLK preparation	Spike surrogate into a premixed solution of 1:1 water:methanol with 0.1% acetic acid	Premixed water:methanol solutions immediately begin to evaporate and do not maintain 1:1 ratio, which affects surrogate recovery.

When applicable, comparability and/or equivalency studies necessary to validate the modification as required per corporate SOP ENV-SOP-CORQ-0011 are retained by local quality personnel for historical reference.

15.0 RESPONSIBILITIES

- All PAS employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement (R&A) in their training file.
- PAS supervisors and managers are responsible for training employees on the procedures in this SOP, implementing the SOP in the work area, and monitoring on-going adherence to the SOP the work area(s) they oversee.
- Local quality personnel are responsible for tracking the currency of the R&A on this SOP for employees at the locations they are assigned to and for notifying the department leaders of overdue assignments.
- All employees of PAS are responsible for following the procedures in this SOP. Unauthorized deviations or departures from this SOP are not allowed except with documented approval from the local Quality Manager and only when those deviations do not violate the Pace® Code of Ethics or Professional Conduct (COR-POL-0004) or associated policy and procedure(s). Hand-edits or manual change to the SOP are not permitted. If a change is desired or necessary, employees must follow the procedures for document revision specified in corporate SOPs ENV-SOP-CORQ-0015, *Document Managemen*t and ENV-SOP-CORQ-0016, *SOP for Creation of SOP and SWI*.
- Local quality personnel are responsible for monitoring conformity to this SOP during routine internal audits of work areas that utilize this SOP and for communicating gaps and deviations found during monitoring to the work area supervisor, who is responsible for correction of the situation.

16.0 ATTACHMENTS

- Appendix A: Targe Analyte Limits, Analytical Parameters, and Calibration
- Appendix B: QC Summary

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17.0 REFERENCES

Note: Where reference exclude a date or edition, the latest edition of the referenced document adopted/recognized by the laboratory's accreditation bodies applies. Refer to the Laboratory Quality Manual [QAMP ENV-MAN-WCOL-0001] for details.

- ENV-SOP-CORQ-0006, *Manual Integration*, current version.
- ENV-SOP-CORQ-0011, *Method Validation*, current version.
- ENV-SOP-CORQ-0015, *Document Management*, current version.
- ENV-SOP-CORQ-0016, *SOP for SOP and SWI,* current version.
- ENV-TMP-CORQ-0007, *Quality Manual Template*, current version.
- COR-POL-0004, *Code of Ethics and Professional Conduct*, current version.
- COR-MAN-001, *Pace® Safety Manua*l, current version.
- ENV-MAN-WCOL-0001, *Laboratory Quality Manual*, current version.
- ASTM Method D8421-22, *Standard Test Method for Determination of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Matrices by Co-solvation followed by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS)*, ASTM International, December 2022.
- SW-846 Test Method 8327, *Per- and Polyfluoroalkyl Substances (PFAS) by Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)*, USEPA, July 2021.
- ASTM Method D7968 – 17a, *Standard Test Method for Determination of Polyfluorinated Compounds in Soil by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS),* ASTM International, 2017

18.0 REVISION HISTORY

Revisions Made from Prior Version

Section	Description of Change
Section 7.2	Updated supplies to what is currently being used.
Section 8.1	Updated reagent catalog numbers to reflect what is currently being used.
Section 8.2	Updated standards to match current practice.
Section 9.3.2	Updated aqueous sample process to reflect current use of 2 surrogate solutions.
Section 9.4.1	Updated analytical sequence for CCV from L5 to L8 to reflect current practice.
Section 9.4.2	Added daily isomer check injection
Section 14.0	Added modifications for the additions of water and methanol to be separate instead of pre-mixed solution.
Table 1	Updated solid LOQ where applicable.

Document Succession: This version replaces the following documents:

Document Number & Version	Document Title	Effective Date:
ENV-SOP-WCOL-0164 v01	PFAS by D8421	5/3/2023

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Appendix A: Target Analyte List and LOQ

Table 1: Routine Analyte List and Limits of Quantitation (LOQ)[1]

Analyte	Aqueous (ng/L)	Solid (ng/g)
PFBA	50	0.5
PFPeA	50	0.5
PFHxA	10	0.1
PFHpA	10	0.1
PFOA	10	0.1
PFNA	10	0.1
PFDA	10	0.1
PFUnA	10	0.1
PFDoA	10	0.1
PFTrDA	10	0.1
PFTeDA[2]	50	0.5
PFBS	10	0.1
PFPeS	10	0.1
PFHxS	10	0.1
PFHpS	10	0.1
PFOS	10	0.1
PFNS	10	0.1
PFDS	10	0.1
PFDoS[2]	50	0.5
4:2 FTS	10	0.1
6:2 FTS	20	0.2
8:2 FTS	10	0.1
PFOSA	10	0.1
NMeFOSA	10	0.1
NEtFOSA	10	0.1
NMeFOSAA	10	0.1
NEtFOSAA	10	0.1
NMeFOSE	10	0.1
NEtFOSE	10	0.1
HFPO-DA	10	0.1
ADONA	10	0.1
PFEESA	10	0.1
PFMPA	10	0.1
PFMBA	10	0.1
NFDHA	10	0.1
9Cl-PF3ONS	10	0.1
11Cl-PF3OUdS	10	0.1
3:3 FTCA	40	0.4
5:3 FTCA	40	0.4
7:3 FTCA	40	0.4
PFPrA	200	2
FHUEA	10	0.1
FOUEA	10	0.1
HQ-115	10	0.1

[1] Values in place as of effective date of this SOP and are based on nominal sample volume/mass. LOQ are subject to change. For the most up to date LOQ, refer to the LIMS or contact the laboratory.

[2] PFTeDA and PFDoS will be reported to listed LOQ; no MDL values will be reported for these compounds.

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Table 2: Analytical Information for Target Analytes and Quantitation Standards

Compound Name	CAS Number	Compound Abbreviation	Parent Ion Mass	Quantification Ion Mass	Confirmation Ion Mass	Quantification Reference Compound
Perfluoropropanoic acid	422-64-0	PFPrA	162.9	118.9	NA	13C4-PFBA
Perfluorobutanoic acid	375-22-4	PFBA	212.9	168.9	NA	13C4-PFBA
Perfluoropentanoic acid	2706-90-3	PFPeA	262.9	218.9	NA	13C5-PFPeA
Perfluorohexanoic acid	307-24-4	PFHxA	312.9	269	118.9	13C5-PFHxA
Perfluoroheptanoic acid	375-85-9	PFHpA	362.9	318.9	168.9	13C4-PFHpA
Perfluorooctanoic acid	335-67-1	PFOA	412.9	369	168.9	13C8-PFOA
Perfluorononanoic acid	375-95-1	PFNA	462.9	419	218.9	13C9-PFNA
Perfluorodecanoic acid	335-76-2	PFDA	512.9	469	218.9	13C6-PFDA
Perfluoroundecanoic acid	2058-94-8	PFUnA	562.9	519	269	13C7-PFUnA
Perfluorododecanoic acid	307-55-1	PFDoA	612.9	568.9	168.9	13C2-PFDoA
Perfluorotridecanoic acid	72629-94-8	PFTrDA	663.0	618.9	168.9	13C2-PFDoA
Perfluorotetradecanoic acid	376-06-7	PFTeDA	712.9	668.9	168.9	13C2-PFTeDA
Perfluorobutanesulfonic acid	375-73-5	PFBS	298.9	79.9	98.9	13C3-PFBS
Perfluoropentansulfonic acid	2706-91-4	PFPeS	348.9	79.9	98.9	13C3-PFHxS
Perfluorohexanesulfonic acid	355-46-4	PFHxS	398.9	79.9	98.9	13C3-PFHxS
Perfluoroheptanesulfonic acid	375-92-8	PFHpS	448.9	79.9	98.9	13C3-PFHxS
Perfluorooctanesulfonic acid	1763-23-1	PFOS	498.9	79.9	98.9	13C8-PFOS
Perfluorononanesulfonic acid	68259-12-1	PFNS	548.9	79.9	98.9	13C8-PFOS
Perfluorodecanesulfonic acid	335-77-3	PFDS	598.9	79.9	98.9	13C8-PFOS
Perfluorododecanesulfonic acid	79780-39-5	PFDoS	698.9	79.9	98.9	13C8-PFOS
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	757124-72-4	4:2FTS	326.9	306.9	80.9	13C2-4:2FTS
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	27619-97-2	6:2FTS	427	406.9	80.9	13C2-6:2FTS
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	39108-34-4	8:2FTS	526.9	506.9	80.9	13C2-8:2FTS
Perfluorooctanesulfonamide	754-91-6	PFOSA	497.9	77.9	NA	13C8-PFOSA
N-methyl perfluorooctanesulfonamide	31506-32-8	NMeFOSA	511.9	168.9	218.9	D3-NMeFOSA
N-ethyl perfluorooctanesulfonamide	4151-50-2	NEtFOSA	525.9	168.9	218.9	D5-NEtFOSA
N-methyl perfluorooctanesulfonamidoacetic acid	2355-31-9	NMeFOSAA	569.9	419	482.9	D3-NMeFOSAA
N-ethyl perfluorooctanesulfonamidoacetic acid	2991-50-6	NEtFOSAA	584	419	482.9	D5-NEtFOSAA

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Compound Name	CAS Number	Compound Abbreviation	Parent Ion Mass	Quantification Ion Mass	Confirmation Ion Mass	Quantification Reference Compound
N-methyl perfluorooctanesulfonamidoethanol	24448-09-7	NMeFOSE	616	58.9	NA	D7-NMeFOSE
N-ethyl perfluorooctanesulfonamidoethanol	1691-99-2	NEtFOSE	630	58.9	NA	D9-NEtFOSE
Hexafluoropropylene oxide dimer acid (GenX)	13252-13-6	HFPO-DA	285	184.9	168.9	13C3-HFPODA
4,8-Dioxa-3H-perfluorononanoic acid	919005-14-4	ADONA	376.9	251	84.9	13C3-PFHxS
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1	9Cl-PF3ONS	530.9	350.9	352.9	13C8-PFOS
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9	11Cl-PF3OUdS	630.8	450.9	452.9	13C8-PFOS
3-Perfluoropropyl propanoic acid (FPrPA)	356-02-5	3:3FTCA	241	177	117	13C5-PFPeA
2H,2H,3H,3H-Perfluorooctanoic acid (FPePA)	914637-49-3	5:3FTCA	340.9	216.9	237	13C5-PFHxA
3-Perfluoroheptyl propanoic acid (FHpPA)	812-70-4	7:3FTCA	440.9	337	316.9	13C5-PFHxA
Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7	PFEESA	314.9	134.9	82.9	13C3-PFBS
Perfluoro-3-methoxypropanoic acid	377-73-1	PFMPA	228.9	84.9	NA	13C4-PFBA
Perfluoro-4-methoxybutanoic acid	863090-89-5	PFMBA	278.9	84.9	NA	13C5-PFPeA
Nonafluoro-3,6-dioxaheptanoic acid	151772-58-6	NFDHA	295	200.9	84.9	13C5-PFHxA
2H-Perfluoro-2-octenoic acid	70887-88-6	FHUEA	356.9	2932.9	NA	13C4-PFHpA
2H-Perfluoro-2-decenoic acid	70887-70-4	FOUEA	456.9	393	NA	13C9-PFNA
Bis(trifluoromethane)sulfonimide	90076-65-6	HQ-115	279.9	146.9	210.9	13C2-6:2 FTS
Labeled Quantitation Standard (SUR) Compounds						
Perfluoro-n-[13C4] butanoic acid	N/A	13C4-PFBA	216.9	171.9	NA	NA
Perfluoro-n-[13C5] pentanoic acid	N/A	13C5-PFPeA	267.9	222.9	NA	NA
Perfluoro-n-[1,2,3,4,6-13C5] hexanoic acid	N/A	13C5-PFHxA	317.9	272.9	NA	NA
Perfluoro-n-[1,2,3,4-13C4] heptanoic acid	N/A	13C4-PFHpA	366.9	321.9	NA	NA
Perfluoro-n-[13C8] octanoic acid	N/A	13C8-PFOA	421	376	NA	NA
Perfluoro-n-[13C9] nonanoic acid	N/A	13C9-PFNA	471.9	426.9	NA	NA
Perfluoro-n-[1,2,3,4,5,6-13C6] decanoic acid	N/A	13C6-PFDA	518.9	473.9	NA	NA
Perfluoro-n-[1,2,3,4,5,6,7-13C7] undecanoic acid	N/A	13C7-PFUnA	569.9	524.9	NA	NA
Perfluoro-n-[1,2-13C2] dodecanoic acid	N/A	13C2-PFDoA	614.9	569.9	NA	NA
Perfluoro-n-[13C2] tetradecanoic acid	N/A	13C2-PFTeDA	714.9	669.9	NA	NA

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Compound Name	CAS Number	Compound Abbreviation	Parent Ion Mass	Quantification Ion Mass	Confirmation Ion Mass	Quantification Reference Compound
Perfluoro-1-[13C3] butanesulfonic acid	N/A	13C3-PFBS	301.9	79.9	NA	NA
Perfluoro-1-[1,2,3-13C3] hexanesulfonic acid	N/A	13C3-PFHxS	401.9	79.9	NA	NA
Perfluoro-1-[13C8] octanesulfonic acid	N/A	13C8-PFOS	506.9	79.9	NA	NA
1H,1H,2H,2H-Perfluoro-1-[1,2-13C2] hexanesulfonic acid	N/A	13C2-4:2FTS	328.9	80.9	309	NA
1H,1H,2H,2H-Perfluoro-1-[1,2-13C2] octanesulfonic acid	N/A	13C2-6:2FTS	428.9	80.9	409	NA
1H,1H,2H,2H-Perfluoro-1-[1,2-13C2] decanesulfonic acid	N/A	13C2-8:2FTS	528.9	80.9	509	NA
Perfluoro-1-[13C8] octanesulfonamide	N/A	13C8-PFOSA	505.9	77.9	NA	NA
N-methyl-d3-perfluoro-1-octanesulfonamide	N/A	D3-NMeFOSA	514.9	168.9	NA	NA
N-ethyl-d5-perfluoro-1-octanesulfonamide	N/A	D5-NEtFOSA	531	168.9	NA	NA
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	N/A	D3-NMeFOSAA	572.9	418.9	NA	NA
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	N/A	D5-NEtFOSAA	589	418.9	NA	NA
N-methyl-d7-perfluorooctanesulfonamidoethanol	N/A	D7-NMeFOSE	623	58.9	NA	NA
N-ethyl-d9 -perfluorooctanesulfonamidoethanol	N/A	D9-NEtFOSE	639	58.9	NA	NA
Tetrafluoro-2-heptafluoropropoxy-13C3 -propanoic acid	N/A	13C3-HFPO-DA	287	168.9	NA	NA

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Table 3: Calibration Standard Concentration (ng/L)

Compound	L1	L2	L3	L4	L5	L6	L7	L8 (CCV)[1]	L9	L10
PFPrA	25.1	50.2	100.4	251	502	1004	2510	5020	7530	10040
PFBA	25.3	50.5	101	252.5	505	1010	2525	5050	7575	10100
PFPeA	25.1	50.3	100.5	251.3	502.5	1005	2512.5	5025	7537.5	10050
PFHxA	5.1	10.1	20.3	50.7	101.4	202.8	507	1014	1521	2028
PFHpA	5	9.9	19.8	49.6	99.2	198.4	496	992	1488	1984
PFOA	5	10	20	50.1	100.2	200.4	501	1002	1503	2004
PFNA	5	10	20	50.1	100.2	200.4	501	1002	1503	2004
PFDA	5	10	20	50	100	200	500	1000	1500	2000
PFUnA	5	10.1	20.2	50.4	100.8	201.6	504	1008	1512	2016
PFDoA	5	10.1	20.2	50.4	100.8	201.6	504	1008	1512	2016
PFTrDA	5	10	20	50.1	100.2	200.4	501	1002	1503	2004
PFTeDA	5.1	10.1	20.3	50.6	101.3	202.5	506.3	1012.5	1518.8	2025
PFBS	5.1	10.1	20.3	50.7	101.4	202.8	507	1014	1521	2028
PFPeS	5	10	20	49.9	99.8	199.6	499	998	1497	1996
PFHxS	5	10.1	20.1	50.3	100.6	201.2	503	1006	1509	2012
PFHpS	4.9	9.9	19.8	49.4	98.8	197.6	494	988	1482	1976
PFOS	5	10	20.1	50.2	100.3	200.6	501.5	1003	1504.5	2006
PFNS	5.2	10.5	20.9	52.3	104.6	209.1	522.8	1045.5	1568.3	2091
PFDS	5	10	20.1	50.2	100.3	200.6	501.5	1003	1504.5	2006
PFDoS	5.1	10.3	20.6	51.5	102.9	205.8	514.5	1029	1543.5	2058
4:2FTS	5.1	10.2	20.4	50.9	101.8	203.6	509	1018	1527	2036
6:2FTS	5	10	20	50.1	100.2	200.3	500.8	1001.5	1502.3	2003
8:2FTS	5.1	10.2	20.4	51	102.1	204.1	510.3	1020.5	1530.8	2041
PFOSA	5.1	10.1	20.2	50.5	101	202	505	1010	1515	2020
NMeFOSA	5.1	10.1	20.2	50.6	101.1	202.2	505.5	1011	1516.5	2022
NEtFOSA	5.1	10.2	20.4	50.9	101.8	203.5	508.8	1017.5	1526.3	2035
NMeFOSAA	5	10	20.1	50.2	100.4	200.8	502	1004	1506	2008
NEtFOSAA	5	10.1	20.2	50.4	100.8	201.6	504	1008	1512	2016
NMeFOSE	5.1	10.1	20.2	50.6	101.2	202.4	506	1012	1518	2024
NEtFOSE	5.1	10.2	20.3	50.8	101.6	203.2	508	1016	1524	2032
HFPO-DA	5	10	20	50.1	100.1	200.2	500.5	1001	1501.5	2002
ADONA	5	10.1	20.1	50.3	100.6	201.2	503	1006	1509	2012
9Cl-PF3ONS	5.2	10.5	21.0	52.4	104.9	209.7	524.3	1048.5	1572.8	2097
11Cl-PF3OUdS	5.2	10.4	20.9	52.2	104.4	208.7	521.8	1043.5	1565.3	2087
3:3FTCA	5	9.9	19.9	49.7	99.4	198.8	497	994	1491	1988
5:3FTCA	5	10	20	50.1	100.2	200.4	501	1002	1503	2004
7:3FTCA	5.1	10.1	20.2	50.5	101	202	505	1010	1515	2020

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Compound	L1	L2	L3	L4	L5	L6	L7	L8 (CCV)[1]	L9	L10
PFEESA	5.1	10.1	20.3	50.7	101.4	202.7	506.8	1013.5	1520.3	2027
PFMPA	5.1	10.2	20.3	50.8	101.6	203.2	508	1016	1524	2032
PFMBA	4.9	9.8	19.6	49.1	98.2	196.4	491	982	1473	1964
NFDHA	5	10	20.1	50.2	100.4	200.8	502	1004	1506	2008
FHUEA	5.1	10.1	20.2	50.6	101.1	202.2	505.5	1011	1516.5	2022
FOUEA	5	10.1	20.1	50.3	100.6	201.2	503	1006	1509	2012
HQ-115	5	10	20	50	99.9	199.8	499.5	999	1498.5	1998
Labeled Quantitation Standard (SUR) Compounds										
13C4-PFBA	160	160	160	160	160	160	160	160	160	160
13C5-PFPeA	160	160	160	160	160	160	160	160	160	160
13C5-PFHxA	160	160	160	160	160	160	160	160	160	160
13C4-PFHpA	160	160	160	160	160	160	160	160	160	160
13C8-PFOA	160	160	160	160	160	160	160	160	160	160
13C9-PFNA	160	160	160	160	160	160	160	160	160	160
13C6-PFDA	160	160	160	160	160	160	160	160	160	160
13C7-PFUnA	160	160	160	160	160	160	160	160	160	160
13C2-PFDoA	160	160	160	160	160	160	160	160	160	160
13C2-PFTeDA	160	160	160	160	160	160	160	160	160	160
13C3-PFBS	160	160	160	160	160	160	160	160	160	160
13C3-PFHxS	160	160	160	160	160	160	160	160	160	160
13C8-PFOS	160	160	160	160	160	160	160	160	160	160
13C2-4:2FTS	160	160	160	160	160	160	160	160	160	160
13C2-6:2FTS	160	160	160	160	160	160	160	160	160	160
13C2-8:2FTS	160	160	160	160	160	160	160	160	160	160
13C8-PFOSA	160	160	160	160	160	160	160	160	160	160
D3-NMeFOSA	160	160	160	160	160	160	160	160	160	160
D5-NEtFOSA	160	160	160	160	160	160	160	160	160	160
D3-NMeFOSAA	160	160	160	160	160	160	160	160	160	160
D5-NEtFOSAA	160	160	160	160	160	160	160	160	160	160
D7-NMeFOSE	160	160	160	160	160	160	160	160	160	160
D9-NEtFOSE	160	160	160	160	160	160	160	160	160	160
13C3-HFPODA	160	160	160	160	160	160	160	160	160	160

[1] *This calibration point is used as the calibration verification (CCV)*

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Appendix B: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Mass Calibration	Annually and on as-needed basis.	Must meet manufacturer's acceptance criteria.	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
Initial Calibration (ICAL)	At instrument set up, after CCV failure and/or major maintenance.	Must meet one of curve fit options presented in Section 9.2.4.	Identify and correct source of problem, repeat	None. Do not proceed with analysis
Initial Calibration Verification (ICV)	After Each ICAL	All analytes must be within ± 30% of the true value. (%R)	Identify and correct source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
RT Window Position	Once per ICAL.	Position is set using the mid-point of the ICAL	NA	NA
RT Window Study	At method set-up and after major instrument maintenance	RT Window is ± 12 secs from RT position	NA	NA
Continuing Calibration Verification (CCV)	Daily, before sample analysis, after every 10 samples, and at end of analytical window.	All native and isotopically labelled compounds within ± 30% recovery.	See Section 9.2.4.4 for required corrective actions based on circumstance.	Qualify analytes with CCV out of criteria.
Qualitative Isomer Check	After each ICAL	All required branched isomers present and visibly resolved from linear peak.	NA	NA
Instrument Blank (IBLK / CCB)	Daily prior to analysis and after high standards, including CCV.	Must meet criteria specified in Section 11.2.1: all detections ≤1/2 LOQ.	Identify and correct source of contamination or performance issue. Reanalyze IBLK.	No samples shall be analyzed until IBLK has met acceptance criteria.
Surrogate (SUR, Labeled Quantitation Standards)	Every field sample, standard and QC sample.	Must meet criteria specified in Section 11.3. Preliminary acceptance range: 70-130% REC (based on calculated concentration).	If batch QC is acceptable, reanalyze to confirm. If confirmed, reprep and reanalyze samples. If reprep is within acceptance, report reprep data. If failure is confirmed by reprep, qualify as matrix impacted.	Qualify outages and explain in case narrative.
Method Blank (MB) / Reagent Blank (MB2)	1 per batch of 20 or fewer samples.	Must meet criteria specified in Section 11.1.1	If IBLK is acceptable, reanalyze MB to confirm. If confirmed, reprep and reanalyze associated impacted samples (if sufficient sample remains). If insufficient sample remains for reprep, narrate and report data associated to	Qualify outages and explain in case narrative.

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QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
			unacceptable MB. See Section 11.1.1 for more details.	
Laboratory Control Sample (LCS)	1 pair per batch of 20 or fewer samples.	70-130% recovery; must meet criteria specified in Section 11.1.2.	If most recent CCV is acceptable, reanalyze LCS to confirm. If low-failure results are confirmed, reprep and reanalyze associated samples (if sufficient sample remains). If insufficient sample remains for reprep, narrate and report data associated to low-failure LCS. If high-failure results are confirmed and sample(s) is ND for failing compound, narrate and report sample data.	Qualify outages and explain in case narrative.
Method Reporting Limit Control Sample (MRLCS)	1 pair per batch of 20 or fewer samples.	50-150% recovery; must meet criteria specified in Section 11.1.2.	If most recent CCV is acceptable, reanalyze MRLCS to confirm. If low-failure results are confirmed, reprep and reanalyze associated samples (if sufficient sample remains). If insufficient sample remains for reprep, narrate and report data associated to low-failure MRLCS. If high-failure results are confirmed and sample(s) is ND for failing compound, narrate and report sample data.	Qualify outages and explain in case narrative.
MS/MSD	Typically, 1 pair per batch of 20 or fewer samples, if possible.	RSD ≤30% between MS/MSD	If possible, reprep to confirm.	Qualify outages and explain in case narrative.
Sample Duplicate (DUP)	1 per batch of 20 or fewer samples, if possible.	RSD ≤30% between parent and DUP	If possible, reprep to confirm.	Qualify outages and explain in case narrative.

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SAP Worksheet #19/30
Analytical SOP
Solid Matrix

Matrix	Analytical Group	Analytical and Preparation Method/ SOP Reference	Containers (number, size, and type)	Sample Volume	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Solid	PFAS	D8421/ D7968/ ENV-SOP-WCOL-0164	90 mL PP	50 grams	<6°C	28 days

SAP Worksheet #23
Analytical SOP Requirements Table
Solid Matrix

Lab SOP Number#	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument/ Equipment	Organization Performing Analysis	Modified for Project Work? (Y/N)
1	ENV-SOP-WCOL-0164	Screening	PFAS/ Non-Potable Water and Solids	Sciex 5500 Triple Quad LC-MS/MS	Pace West Columbia, SC	N

SAP Worksheet #24
Analytical Instrument Calibration Table
Solid Matrix

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS/MS	Mass Calibration	Instrument must have a valid mass calibration prior to any sample analysis. Mass calibration is verified after each mass calibration, prior to initial calibration (ICAL).	Calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for every acquisition in an analytical run. Mass calibration must be verified to be ±0.5 amu of the true value, by acquiring a full scan continuum mass spectrum of a PFAS stock standard.	If the mass calibration fails, then recalibrate. If it fails again, consult manufacturer instructions on corrective maintenance.	Analyst, supervisor	1

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS/MS	Calibration, Calibration Verification and Spiking Standards	All analytes. Note: Standards containing both branched and linear isomers are to be used during method validation and when reestablishing retention times, to ensure the total response is quantitated for that analyte. Technical grade standards cannot be used for quantitative analysis.	PFAS method analytes may consist of both branched and linear isomers, but quantitative standards that contain the linear and branched isomers do not exist for all method analytes. For PFAS that do not have a quantitative branched and linear standard, identify the branched isomers by analyzing a qualitative standard that includes both linear and branched isomers and determine retention times, transitions and transition ion ratios. Quantitate samples by integrating the total response (i.e., accounting for peaks that are identified as linear and branched isomers) and relying on the initial calibration that uses the linear isomer quantitative standard.	NA	Analyst, supervisor	1
LC-MS/MS	Mass Spectral Acquisition Rate	Each analyte, extracted internal standard (EIS) analyte.	A minimum of 10 spectra scans should be acquired across each chromatographic peak.	NA	Analyst, supervisor	1

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS/MS	Initial calibration (ICAL); Calibration can be linear (minimum of 5 standards); weighting is allowed.	Prior to sample analysis: at instrument set-up and after major maintenance and/or ICV or CCV failure.	ICAL must meet one of the two options below: Option 1: The %RSD of the RFs for all analytes must be ≤30%. Option 2: Linear or non-linear calibrations must have %RSE ≤30% for all compounds. The isotopically labeled analog of an analyte (Extracted Internal Standard Analyte) must be used for quantitation if commercially available (Isotope Dilution Quantitation).	Perform maintenance, if necessary, and repeat calibration if criterion is not met. No samples shall be analyzed until ICAL has passed.	Analyst, supervisor	1
LC-MS/MS	Instrument blank (IBLK)	Immediately following the highest standard analyzed during ICAL and daily, prior to sample analysis.	Concentration of each analyte must be ≤½ the LOQ. Instrument Blank must contain EIS to enable quantitation of contamination.	If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met.	Analyst, supervisor	1

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS/MS	Initial calibration verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Analyte concentrations must be within ± 30% of their true value.	Correct problem and verify second source standard; rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, supervisor	1
LC-MS/MS	Continuing Calibration Verification (CCV)	Daily prior to sample analysis; after every 10 field samples; at end of analytical sequence	All analytes must be within ± 30% of their true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all samples since the last acceptable CCV.	Analyst, supervisor	1

SAP Worksheet #25
Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
Solid Matrix

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
LC-MS/MS	Check column pressure and mobile phase levels/ expirations daily. Perform the following as needed: prepare aqueous mobile phase, clean/replace injection needle, replace guard cartridge, backflush/replace column, replace injector seat, clean curtain/orifice plate, retune MS.	PFAS	Inspect all tubing connections at time of maintenance to assure no leaks present. Monitor instrument performance via calibrations, CCVs, and blanks.	Initially, after major maintenance, CCV not meeting 2X.	Same as initial calibration and continuing calibration verification.	Same as initial calibration and continuing calibration verification.	Analyst/Supervisor	1

SAP Worksheet #28
Laboratory QC Samples Table
Solid Matrix

QC Sample	Frequency/No.	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Sample PFAS Identification	All analytes detected in a sample	A minimum of two ion transitions (Precursor→quant ion and precursor→ confirmation ion) and the ion transitions ratio per analyte are required for confirmation. Exception is made for analytes where two transitions do not exist (PFPrA, PFBA, PFPeA, PFOSA, NMeFOSE, NEtFOSE, PFMPA, PFMBA, FHUEA, and FOUEA). In-house acceptance criteria for evaluation of ion ratios must be used and must not exceed 70-130%. Signal to Noise Ratio (S/N) must be ≥3 for all ions used for quantification and must be ≥3 for all ions used for confirmation.	Any detected quantitation ion peak that does not meet the ion ratio or S/N criteria should be closely reviewed to determine whether the peak should be rejected or not; generally, these peaks should be rejected unless qualitative measures show interference.	Analyst	NA	NA

QC Sample	Frequency/No.	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Retention Time window position establishment	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve.	NA	NA	NA	NA
Retention Time (RT) window width	Every field sample standard, blank, and QC sample.	RT of each analyte and EIS analyte must fall within 0.4 minutes of the predicted retention times from the midpoint standard of the ICAL. Analytes must elute within 0.1 minutes of the associated EIS; this criterion applies only to analyte and labeled analog pairs.	Correct problem and reanalyze samples.	Analyst	NA	NA
Extracted Internal Standard Analytes (EIS, aka SUR)	Every field sample, standard, blank, and QC sample.	Added to aqueous samples prior to preparation. Extracted Internal Standard Analyte recoveries must be within 50% to 150%.	Correct problem. If required, re-prepare and reanalyze affected field and QC samples.	Analyst, supervisor	Apply Q-flag and discuss in the Case Narrative only if reanalysis confirms failures in the same manner.	Failing analytes shall be thoroughly documented in the Case Narrative.

QC Sample	Frequency/No.	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample, whichever is greater.	Correct problem. If required, re-prepare and reanalyze MB and all QC samples and field samples processed with the contaminated blank. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, supervisor	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid MB. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

QC Sample	Frequency/No.	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One per preparatory batch.	Blank spiked with all analytes at a concentration near the mid-level calibration concentration. Recoveries must be 50%-150%.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, supervisor	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

QC Sample	Frequency/No.	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Matrix Spike (MS)	One per preparatory batch, if possible and/or requested.	Sample spiked with all analytes at a concentration near the mid-level calibration concentration. Recoveries must be 50%-150%.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, supervisor	For the specific analyte(s) in the parent sample, apply flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD)	One per preparatory batch, if possible and/or requested.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Recoveries must be 50%-150%. RPD ≤ 30% (between MS and MSD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, supervisor	For the specific analyte(s) in the parent sample, apply flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source or difference.

QAPP Worksheet #15 — Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
Matrix: Solids
Analytical Group: PFAS
Analytical SOP: ENV-SOP-WCOL-0164
Analytical Method: ASTM D8421

Analyte	Acronym	CAS Number	Detection Limit (DL) µg/Kg	Limit of Quantitation (LOQ) µg/Kg	Screening Standard and Source	Accuracy Control Limit (%R)	Precision Control Limit RPD
Perfluorobutanoic acid	PFBA	375-22-4	0.01348	0.5		50-150%	≤30%
Perfluoropentanoic acid	PFPeA	2706-90-3	0.03089	0.5		50-150%	≤30%
Perfluorohexanoic acid	PFHxA	307-24-4	0.00354	0.1		50-150%	≤30%
Perfluoroheptanoic acid	PFHpA	375-85-9	0.00972	0.1		50-150%	≤30%
Perfluorooctanoic acid	PFOA	335-67-1	0.01324	0.1		50-150%	≤30%
Perfluorononanoic acid	PFNA	375-95-1	0.00599	0.1		50-150%	≤30%
Perfluorodecanoic acid	PFDA	335-76-2	0.0145	0.1		50-150%	≤30%
Perfluoroundecanoic acid	PFUnA	2058-94-8	0.00785	0.1		50-150%	≤30%
Perfluorododecanoic acid	PFDoA	307-55-1	0.00611	0.1		50-150%	≤30%
Perfluorotridecanoic acid	PFTrDA	72629-94-8	0.00826	0.1		50-150%	≤30%
Perfluorotetradecanoic acid	PFTeDA	376-06-7	0.5 (1)	0.5		50-150%	≤30%
Perfluorobutanesulfonic acid	PFBS	375-73-5	0.00727	0.1		50-150%	≤30%
Perfluoropentansulfonic acid	PFPeS	2706-91-4	0.00663	0.1		50-150%	≤30%
Perfluorohexanesulfonic acid	PFHxS	355-46-4	0.00445	0.1		50-150%	≤30%
Perfluoroheptanesulfonic acid	PFHpS	375-92-8	0.00631	0.1		50-150%	≤30%

Analyte	Acronym	CAS Number	Detection Limit (DL) µg/Kg	Limit of Quantitation (LOQ) µg/Kg	Screening Standard and Source	Accuracy Control Limit (%R)	Precision Control Limit RPD
Perfluorooctanesulfonic acid	PFOS	1763-23-1	0.00397	0.1		50-150%	≤30%
Perfluorononanesulfonic acid	PFNS	68259-12-1	0.00465	0.1		50-150%	≤30%
Perfluorodecanesulfonic acid	PFDS	335-77-3	0.00618	0.1		50-150%	≤30%
Perfluorododecanesulfonic acid	PFDoS	79780-39-5	0.5 (1)	0.5		50-150%	≤30%
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	4:2FTS	757124-72-4	0.01008	0.1		50-150%	≤30%
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	6:2FTS	27619-97-2	0.03453	0.2		50-150%	≤30%
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	8:2FTS	39108-34-4	0.02314	0.1		50-150%	≤30%
Perfluorooctanesulfonamide	PFOSA	754-91-6	0.00308	0.1		50-150%	≤30%
N-methyl perfluorooctanesulfonamide	NMeFOSA	31506-32-8	0.00576	0.1		50-150%	≤30%
N-ethyl perfluorooctanesulfonamide	NEtFOSA	4151-50-2	0.00421	0.1		50-150%	≤30%
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9	0.01052	0.1		50-150%	≤30%
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6	0.01288	0.1		50-150%	≤30%
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7	0.01259	0.1		50-150%	≤30%
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1691-99-2	0.00377	0.1		50-150%	≤30%
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6	0.00397	0.1		50-150%	≤30%
4,8-Dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4	0.0052	0.1		50-150%	≤30%
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1	0.0034	0.1		50-150%	≤30%

Analyte	Acronym	CAS Number	Detection Limit (DL) µg/Kg	Limit of Quantitation (LOQ) µg/Kg	Screening Standard and Source	Accuracy Control Limit (%R)	Precision Control Limit RPD
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5	0.00314	0.1		50-150%	≤30%
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6	0.00794	0.1		50-150%	≤30%
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	756426-58-1	0.0036	0.1		50-150%	≤30%
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9	0.00433	0.1		50-150%	≤30%
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7	0.00458	0.1		50-150%	≤30%
3-Perfluoropropyl propanoic acid	3:3FTCA	356-02-5	0.01356	0.4		50-150%	≤30%
2H,2H,3H,3H-Perfluorooctanoic acid	5:3FTCA	914637-49-3	0.01782	0.4		50-150%	≤30%
3-Perfluoroheptyl propanoic acid	7:3FTCA	812-70-4	0.01265	0.4		50-150%	≤30%
Pentafluorpropanoic acid	PFPrA	422-64-0	0.06142	2		50-150%	≤30%
2H-perfluoro-2-octenoic acid	FHUEA	70887-88-6	0.01092	0.1		50-150%	≤30%
2H-perfluoro-2-decenoic acid	FOUEA	70887-84-2	0.01114	0.1		50-150%	≤30%
Bis(trifluoromethane)sulfonimide	HQ-115	90076-65-6	0.00437	0.1		50-150%	≤30%

Note (1): An MDL for this compound could not be established. As such, the laboratory will report to the LOQ.

QAPP Worksheet #15 — Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
Matrix: Non-Potable Water
Analytical Group: PFAS
Analytical SOP: ENV-SOP-WCOL-0164
Analytical Method: ASTM D8421

Analyte	Acronym	CAS Number	Detection Limit (DL) ng/L	Limit of Quantitation (LOQ) ng/L	Screening Standard and Source	Accuracy Control Limit (%R)	Precision Control Limit RPD
Perfluorobutanoic acid	PFBA	375-22-4	8.3	50		70-130%	≤30%
Perfluoropentanoic acid	PFPeA	2706-90-3	14	50		70-130%	≤30%
Perfluorohexanoic acid	PFHxA	307-24-4	1.4	10		70-130%	≤30%
Perfluoroheptanoic acid	PFHpA	375-85-9	3.2	10		70-130%	≤30%
Perfluorooctanoic acid	PFOA	335-67-1	4.1	10		70-130%	≤30%
Perfluorononanoic acid	PFNA	375-95-1	2.2	10		70-130%	≤30%
Perfluorodecanoic acid	PFDA	335-76-2	2.2	10		70-130%	≤30%
Perfluoroundecanoic acid	PFUnA	2058-94-8	1.4	10		70-130%	≤30%
Perfluorododecanoic acid	PFDoA	307-55-1	2.0	10		70-130%	≤30%
Perfluorotridecanoic acid	PFTrDA	72629-94-8	5.8	10		70-130%	≤30%
Perfluorotetradecanoic acid	PFTeDA	376-06-7	50(1)	50		50-150%	≤30%
Perfluorobutanesulfonic acid	PFBS	375-73-5	3.5	10		70-130%	≤30%
Perfluoropentansulfonic acid	PFPeS	2706-91-4	2.9	10		70-130%	≤30%
Perfluorohexanesulfonic acid	PFHxS	355-46-4	1.6	10		70-130%	≤30%
Perfluoroheptanesulfonic acid	PFHpS	375-92-8	1.6	10		70-130%	≤30%

Analyte	Acronym	CAS Number	Detection Limit (DL) ng/L	Limit of Quantitation (LOQ) ng/L	Screening Standard and Source	Accuracy Control Limit (%R)	Precision Control Limit RPD
Perfluorooctanesulfonic acid	PFOS	1763-23-1	1.6	10		70-130%	≤30%
Perfluorononanesulfonic acid	PFNS	68259-12-1	1.9	10		70-130%	≤30%
Perfluorodecanesulfonic acid	PFDS	335-77-3	2.6	10		70-130%	≤30%
Perfluorododecanesulfonic acid	PFDoS	79780-39-5	50(1)	50		50-150%	≤30%
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	4:2FTS	757124-72-4	1.9	10		70-130%	≤30%
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	6:2FTS	27619-97-2	4.4	20		70-130%	≤30%
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	8:2FTS	39108-34-4	5.2	10		70-130%	≤30%
Perfluorooctanesulfonamide	PFOSA	754-91-6	1.6	10		70-130%	≤30%
N-methyl perfluorooctanesulfonamide	NMeFOSA	31506-32-8	2.1	10		70-130%	≤30%
N-ethyl perfluorooctanesulfonamide	NEtFOSA	4151-50-2	2.6	10		70-130%	≤30%
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9	2.7	10		70-130%	≤30%
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6	3.5	10		70-130%	≤30%
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7	4.5	10		70-130%	≤30%
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1691-99-2	2.6	10		70-130%	≤30%
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6	2.4	10		70-130%	≤30%
4,8-Dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4	1.7	10		70-130%	≤30%
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1	1.4	10		70-130%	≤30%

Analyte	Acronym	CAS Number	Detection Limit (DL) ng/L	Limit of Quantitation (LOQ) ng/L	Screening Standard and Source	Accuracy Control Limit (%R)	Precision Control Limit RPD
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5	2.0	10		70-130%	≤30%
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6	1.8	10		70-130%	≤30%
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	756426-58-1	1.8	10		70-130%	≤30%
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9	3.0	10		70-130%	≤30%
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7	1	10		70-130%	≤30%
3-Perfluoropropyl propanoic acid	3:3FTCA	356-02-5	5.0	40		70-130%	≤30%
2H,2H,3H,3H-Perfluorooctanoic acid	5:3FTCA	914637-49-3	4.9	40		70-130%	≤30%
3-Perfluoroheptyl propanoic acid	7:3FTCA	812-70-4	2.7	40		70-130%	≤30%
Pentafluorpropanoic acid	PFPrA	422-64-0	34	200		70-130%	≤30%
2H-perfluoro-2-octenoic acid	FHUEA	70887-88-6	4.6	10		70-130%	≤30%
2H-perfluoro-2-decenoic acid	FOUEA	70887-84-2	3.2	10		70-130%	≤30%
Bis(trifluoromethane)sulfonimide	HQ-115	90076-65-6	3.0	10		70-130%	≤30%

Note (1): An MDL for this compound could not be established. As such, the laboratory will report to the LOQ.

SAP Worksheet #19/30
Analytical SOP Requirements Table
ASTM D8421
Aqueous Matrix

Matrix	Analytical Group	Analytical and Preparation Method/ SOP Reference	Containers (number, size, and type)	Sample Volume	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Aqueous	PFAS	D8421/ ENV-SOP-WCOL-0164	2 X 15 mL HDPE or PP centrifuge tube	5 mL	<6°C	Preparation: 28 days from collection Analysis: 28 days from preparation

QAPP Worksheet #23 — Analytical SOPs
ASTM D8421
Aqueous Matrix

Lab SOP Number#	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument/ Equipment	Organization Performing Analysis	Modified for Project Work? (Y/N)
1	ENV-SOP-WCOL-0164	Screening	PFAS/ Non-Potable Water and Solids	Sciex 5500 Triple Quad LC-MS/MS	Pace West Columbia, SC	N

SAP Worksheet #24
Analytical Instrument Calibration Table
ASTM D8421
Aqueous Matrix

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS/MS	Mass Calibration	Instrument must have a valid mass calibration prior to any sample analysis. Mass calibration is verified after each mass calibration, prior to initial calibration (ICAL).	Calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for every acquisition in an analytical run. Mass calibration must be verified to be ±0.5 amu of the true value, by acquiring a full scan continuum mass spectrum of a PFAS stock standard.	If the mass calibration fails, then recalibrate. If it fails again, consult manufacturer instructions on corrective maintenance.	Analyst, supervisor	1

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS/MS	Calibration, Calibration Verification and Spiking Standards	All analytes. Note: Standards containing both branched and linear isomers are to be used during method validation and when reestablishing retention times, to ensure the total response is quantitated for that analyte. Technical grade standards cannot be used for quantitative analysis.	PFAS method analytes may consist of both branched and linear isomers, but quantitative standards that contain the linear and branched isomers do not exist for all method analytes. For PFAS that do not have a quantitative branched and linear standard, identify the branched isomers by analyzing a qualitative standard that includes both linear and branched isomers and determine retention times, transitions and transition ion ratios. Quantitate samples by integrating the total response (i.e., accounting for peaks that are identified as linear and branched isomers) and relying on the initial calibration that uses the linear isomer quantitative standard.	NA	Analyst, supervisor	1
LC-MS/MS	Mass Spectral Acquisition Rate	Each analyte, extracted internal standard (EIS) analyte.	A minimum of 10 spectra scans should be acquired across each chromatographic peak.	NA	Analyst, supervisor	1

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS/MS	Initial calibration (ICAL); Calibration can be linear (minimum of 5 standards); weighting is allowed.	Prior to sample analysis: at instrument set-up and after major maintenance and/or ICV or CCV failure.	ICAL must meet one of the two options below: Option 1: The %RSD of the RFs for all analytes must be ≤30%. Option 2: Linear or non-linear calibrations must have %RSE ≤30% for all compounds. The isotopically labeled analog of an analyte (Extracted Internal Standard Analyte) must be used for quantitation if commercially available (Isotope Dilution Quantitation).	Perform maintenance, if necessary, and repeat calibration if criterion is not met. No samples shall be analyzed until ICAL has passed.	Analyst, supervisor	1
LC-MS/MS	Instrument blank (IBLK)	Immediately following the highest standard analyzed during ICAL and daily, prior to sample analysis.	Concentration of each analyte must be ≤½ the LOQ. Instrument Blank must contain EIS to enable quantitation of contamination.	If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met.	Analyst, supervisor	1

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
LC-MS/MS	Initial calibration verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Analyte concentrations must be within ± 30% of their true value.	Correct problem and verify second source standard; rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, supervisor	1
LC-MS/MS	Continuing Calibration Verification (CCV)	Daily prior to sample analysis; after every 10 field samples; at end of analytical sequence	All analytes must be within ± 30% of their true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all samples since the last acceptable CCV.	Analyst, supervisor	1

SAP Worksheet #25
Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
ASTM D8421
Aqueous Matrix

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
LC-MS/MS	Check column pressure and mobile phase levels/ expirations daily. Perform the following as needed: prepare aqueous mobile phase, clean/replace injection needle, replace guard cartridge, backflush/replace column, replace injector seat, clean curtain/orifice plate, retune MS.	PFAS	Inspect all tubing connections at time of maintenance to assure no leaks present. Monitor instrument performance via calibrations, CCVs, and blanks.	Initially, after major maintenance, CCV not meeting 2X.	Same as initial calibration and continuing calibration verification.	Same as initial calibration and continuing calibration verification.	Analyst/Supervisor	1

SAP Worksheet #28
Laboratory QC Samples Table
ASTM D8421
Aqueous Matrix

QC Sample	Frequency/No.	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Sample PFAS Identification	All analytes detected in a sample	A minimum of two ion transitions (Precursor→quant ion and precursor→ confirmation ion) and the ion transitions ratio per analyte are required for confirmation. Exception is made for analytes where two transitions do not exist (PFPrA, PFBA, PFPeA, PFOSA, NMeFOSE, NEtFOSE, PFMPA, PFMBA, FHUEA, and FOUEA). In-house acceptance criteria for evaluation of ion ratios must be used and must not exceed 70-130%. Signal to Noise Ratio (S/N) must be ≥3 for all ions used for quantification and must be ≥3 for all ions used for confirmation.	Any detected quantitation ion peak that does not meet the ion ratio or S/N criteria should be closely reviewed to determine whether the peak should be rejected or not; generally, these peaks should be rejected unless qualitative measures show interference.	Analyst	NA	NA

QC Sample	Frequency/No.	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Retention Time window position establishment	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve.	NA	NA	NA	NA
Retention Time (RT) window width	Every field sample standard, blank, and QC sample.	RT of each analyte and EIS analyte must fall within 0.4 minutes of the predicted retention times from the midpoint standard of the ICAL. Analytes must elute within 0.1 minutes of the associated EIS; this criterion applies only to analyte and labeled analog pairs.	Correct problem and reanalyze samples.	Analyst	NA	NA
Extracted Internal Standard Analytes (EIS, aka SUR)	Every field sample, standard, blank, and QC sample.	Added to aqueous samples prior to preparation. Extracted Internal Standard Analyte recoveries must be within 70% to 130%, except as noted in WS 15 for select compounds.	Correct problem. If required, re-prepare and reanalyze affected field and QC samples.	Analyst, supervisor	Apply Q-flag and discuss in the Case Narrative only if reanalysis confirms failures in the same manner.	Failing analytes shall be thoroughly documented in the Case Narrative.

QC Sample	Frequency/No.	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample, whichever is greater.	Correct problem. If required, re-prepare and reanalyze MB and all QC samples and field samples processed with the contaminated blank. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, supervisor	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid MB. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

QC Sample	Frequency/No.	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One per preparatory batch.	Blank spiked with all analytes at a concentration near the mid-level calibration concentration. Recoveries must be 70%-130%, except as noted in WS 15 for select compounds.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, supervisor	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

QC Sample	Frequency/No.	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Matrix Spike (MS)	One per preparatory batch, if possible and/or requested.	Sample spiked with all analytes at a concentration near the mid-level calibration concentration. Recoveries must be 70%-130% except as noted in WS 15 for select compounds.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, supervisor	For the specific analyte(s) in the parent sample, apply flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).
Matrix Spike Duplicate (MSD)	One per preparatory batch, if possible and/or requested.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. Recoveries must be 70%-130% except as noted in WS 15 for select compounds. RPD ≤ 30% (between MS and MSD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst, supervisor	For the specific analyte(s) in the parent sample, apply flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source or difference.

LAB SUPERVISOR: _____

QA OFFICER: Olga Y. Gaidan

EFFECTIVE DATE: 4-16-2020

TITLE: DETERMINATION OF ORGANOCHLORINE PESTICIDES USING GC SYSTEM
REFERENCES: SW846 8081B (Revision 2, February 2007)
REVISED SECTIONS: 4.0 (MDL),10.1.6.2
DELETED SECTIONS: 3.2.1, 3.2.2, 10.1.6.2.1
ADDED SECTIONS: 10.1.6.3, 10.1.6.4, 10.1.6.5, 10.1.6.6, 10.1.6.7

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the analytical procedures, which are utilized by SGS to acquire samples for analysis of organochlorine pesticides and screening of polychlorinated biphenyls (PCBs) by gas chromatography with Electron Capture Detectors (ECD).

1.2 The method is applicable to extracts from solid and liquid matrices. The compounds listed in Table 1 are determined by a dual-column analysis system.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (from 250 mL to 1 L for liquids, 15 g for solids) is extracted using the appropriate matrix-specific sample extraction technique. Liquid samples are extracted at neutral pH with methylene chloride using Method 3510 (separatory funnel). Solid samples are extracted using Method 3546 Microwave Extraction or Method 3550C, Sonication. A variety of cleanup steps may be applied to the extract, depending on the nature of the matrix interferences and the target analytes. Cleanups include Florisil (Method 3620), silica gel (Method 3630), gel permeation chromatography (Method 3640), and sulfur (Method 3660).

2.2 After cleanup, the extract is analyzed by injecting1-µL of sample **into a gas chromatograph.** The sample is split between two different narrow-bore fused silica capillary columns and run through two electron capture detectors (GC/ECD).

2.3 The peaks detected are qualitatively identified by comparison to retention times specific to the known target list of compounds on two different column types (primary and confirmation).

2.4 If sensitivity permits, the positive hit must be confirmed by GC/MS method 8270D.

2.5 Once identified the compound is quantitated by internal standard techniques with an average response factor generated from a calibration curve.

3.0 REPORTING LIMIT & METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve. RL's may vary depending on matrix and sample volumes or weight and percent moisture. Refer to Table 1 for current reporting limits.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B, Revision 2. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

4.0 DEFINITIONS

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. The different types of blanks are Method Blank, Instrument Blank, Storage Blank, and Sulfur Blank.

FIELD BLANK – an analytical sample prepared from organic-free water and carried through the sampling handling protocol serves as a check for contamination.

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CONTINUING CALIBRATION - analytical standard run every 12 hours and at the end of analytical sequence to verify the initial calibration of the system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - used herein synonymously with the term's continuous extraction, continuous liquid extraction, and liquid extraction. This extraction technique involves boiling the extraction solvent in a flask and condensing the solvent above the aqueous sample. The condensed solvent drips through the sample, extracting the compounds of interest from the aqueous phase.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the electron capture detector to the target compounds.

MATRIX - the predominant material of which the sample to be analyzed is composed. A sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD DETECTION LIMITS (MDLs) – The minimum concentration of a substance that can be measured and reported with 99% confidence that the measured concentration is distinguishable from method blank results.

PERCENT DIFFERENCE (%D) - to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

REAGENT WATER - water in which an interferant is not observed at or above the minimum detection limit of the parameters of interest.

RELATIVE PERCENT DIFFERENCE (RPD) - to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

RELATIVE RESPONSE FACTOR (RRF) - a measure of the instrument response of an analyte. Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

RETENTION TIME (RT) - the time required (in minutes) for a standard compound to elute from a chromatographic column.

SURROGATES - for semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recoveries. Surrogate are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

INSTRUMENT BLANK - a system evaluation sample containing lab reagent grade water with internal standards and/or surrogate standards added. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 The following analytes covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and the BHCs. Primary standards of these toxic compounds must be prepared in a hood. A NIOSH/Mass approved toxic gas respirator must be worn when the analyst handles high concentrations of these toxic compounds.

6.0 INTERFERENCES

6.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

6.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other stages of sample processing. Refer to "The Preparation of Glassware for Extraction of Organic Contaminants" SOP for practices utilized in the extraction department.

6.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary from source to source. Interferences such as sulfur and phthalate are treated with copper and alumina by organics preparation respectively.

6.3.1 The presence of elemental sulfur will result in broad peaks that interfere with detection of early-eluting organochlorine pesticides. Method 3660 is suggested for removal of sulfur.

6.3.2 Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interference from phthalate esters.

6.4 Waxes, lipids, and other high molecular weight materials can be removed by method-3640 (Gel Permeation Chromatography-GPC column cleanup).

6.5 To reduce carryover when high-concentration samples are sequentially analyzed, the syringe must be rinsed out between samples with solvent.

6.6 In the case where an unusually concentrated sample is encountered, it must be followed by the analysis of an instrument blank. An instrument blank is a sample containing hexane with surrogate standards added at 20 ppb. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

7.0 SAMPLE PRESERVATION AND HOLDING TIME

7.1 PRESERVATION

7.1.1 Water Samples

7.1.1.1 Collect samples in 300 mL glass amber bottles without preservatives.

7.1.1.2 A minimum of 250 mL of an unpreserved sample is required for extraction. Additional sample volume is necessary for any samples used for matrix spike and matrix spike duplicates. Therefore, 1 liter of at least one sample in every group of 20 field samples is required for analysis to accommodate all quality control requirements.

7.1.2 Soil Samples

7.1.2.1 Samples are collected in a 300-ml amber glass sample bottle. No preservative is required.

7.1.3 Sample must be taken with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing possible phthalate contamination.

7.1.4 The samples must be protected from light and refrigerated at $\leq 6\ ^{0}C$ from the time of receipt until extraction and analysis.

7.2 HOLDING TIME

7.2.1 Aqueous sample must be extracted within 7 days of sampling.

7.2.2 Soil sample must be extracted within 14 days of sampling.

7.2.3 Extracts must be analyzed within 40 days following extraction.

8.0 APPARATUS AND MATERIALS

8.1 GAS CHROMATOGRAPH SYSTEM

8.1.1 Gas Chromatograph – Agilent or Hewlett Packard Models 7890, 6890 and 5890. The analytical system is completed with a temperature programmable gas chromatograph and all required accessories including syringes, capillary chromatographic columns, and gases. The capillary column is directly coupled to the source. The injection port is designed for splitless injection with capillary columns.

8.1.2 Columns

8.1.2.1 Column pair 1

8.1.2.1.1 30 m x 0.32 mm ID, 0.5 μm film thickness fused silica, DB-1701 narrow-bore capillary column or equivalent.

8.1.2.1.2 30 m x 0.32 mm ID, 0.5 μm film thickness fused silica, DB-5 narrow-bore capillary column.

8.1.2.2 Column pair 2

8.1.2.2.1 30 m x 0.32 mm ID, 0.5 μm film thickness fused silica, RTX CLPI narrow-bore capillary column or equivalent.

8.1.2.2.2 30 m x 0.32 mm ID, 0.25 μm film thickness fused silica, RTX CLPII narrow-bore capillary column or equivalent.

8.1.3 Detectors

8.1.3.1 Electron Capture Detectors (HP).

8.1.3.2 Micro Electron Capture Detectors (HP).

8.2 AUTOSAMPLER

8.2.1 Agilent or Hewlett Packard Model 7673A, 7683, 7643A capable of holding 100 of 2-ml crimp vials.

8.3 DATA SYSTEM

8.3.1 MSD interfaced to the gas chromatograph which allows the continuous acquisition and storage on machine-readable media (disc) of all chromatographic data obtained throughout the duration of the analysis.

8.3.2 The ENVIROQUANT (PC) data system is capable of quantitation using multipoint calibration.

8.3.3 Legato Networker with lookup database on 4mm DAT tape for long term, off line magnetic storage of data.

8.4 SYRINGES

8.4.1 Manually held ul graduated syringes, various volumes (Hamilton or equiv.).

8.4.2 10 µl graduated, auto sampler (Hamilton or equiv.).

8.5 VOLUMETRIC FLASKS, Class A.

9.0 REAGENTS AND STANDARDS

9.1 Refer to SGS Sample Preparation SOPs EOP001 and EOP040A for reagents and standards used for sample extraction.

9.2 Solvents - Ultra pure, chromatography graded Hexane.

9.3 Stock Standard Solutions

9.3.1 Two separate sources of commercially prepared standards with traceability documentation are used.

9.3.1.1 Pesticides Mixtures containing one or more of the following compounds: alpha-BHC, beta-BHC, delta-BHC, gamma-BHC(Lindane), Heptachlor, Aldrin, Heptachlor Epoxide, Endosulfan I, Dieldrin, 4,4'-DDE, Endrin, Endosulfan II, 4,4'-DDD, Endosulfan sulfate, 4,4—DDT, Methoxychlor, Endrin ketone, Endrin Aldehyde, alpha-Chlordane & gamma-chlordane, Alachlor and Hexachlorobenzene.

9.3.1.2 Individual standards containing Toxaphene, Chlordane, 2'4 DDT and Mirex.

9.4 Working Solutions

9.4.1 Prepare working solutions, using stock solution, in hexane, as needed, that contain the compounds of interest, either singly or mixed together. Refer to Table 3 for details.

9.5 Calibration Standards

9.5.1 Initial Calibration Standards

9.5.1.1 Calibration standards are prepared at a minimum of five concentrations, including surrogates, from the above working solutions. Suggested levels and preparations are shown in Table 4A.

9.5.1.2 Separate calibration standards are required for each multi-component target analyte (i.e., Toxaphene and Chlordane). Unless otherwise necessary for a specific project, such as Ohio VAP or the Dept. of Defense (DoD), a single calibration standard near the mid-point of the expected calibration range of each multi-component analyte is employed. Refer to Table 4B and 4C for preparation scheme. Optional curves as shown on Table 4D and 4E may also be used for a multi-point calibration per project's specification.

9.5.2 Continuing Calibration Verification (CCV)

9.5.2.1 Continuing calibration checks containing all the single-component analytes are prepared at concentrations of 10 μg/l, 25 ug/l and 50 μg/l as described in Table 5. During analysis, these alternate concentrations are run to check the initial calibration.

9.5.2.2 In situations where only Toxaphene or Chlordane is of interest for a specific project and for Ohio VAP multi-level calibration checks of each multi-component analyte of interest may be prepared and analyzed throughout the analytical sequence.

9.6 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

9.6.1 Prepare the ICV standards from separate sources of stock standards from the calibration curve following the procedures in Tables 3 and 4.

9.6.2 The ICV must be analyzed immediately following the initial calibration.

9.7 Surrogates

9.7.1 Tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) are used as surrogate standards for this method.

9.7.2 A calibration range must be constructed for the surrogate compounds. Accordingly, appropriate amounts of surrogates are mixed with each calibration solution to define a range similar to the target compounds.

9.7.3 Surrogate compounds are also contained in continuing calibration checks, and second source calibration check standard.

9.7.4 Spike each sample, QC sample and blank with an appropriate amount of corresponding surrogate spiking solution, prior to extraction, for a final concentration in the extract of 40 μg/l of each surrogate compound.

9.8 Internal Standard Solution

9.8.1 1-Bromo-2-nitrobenzene (CAS #577-19-5) is the internal standard being used as recommended for dual column analysis by sw846. The standard is being made at 5 mg/L, and 10uL is being added to all standards and samples for a final concentration of 50ug/L.

9.9 Breakdown Evaluation Solution

9.9.1 The DDT and Endrin breakdown evaluation solution is prepared in hexane as outlined in Table 6.

9.10 Instrument Blank

9.10.1 An instrument blank is run after each Continuing Calibration Check. Spike hexane with an appropriate amount of surrogate spiking solution for a final concentration of 40 μg/l of each surrogate compound.

9.10 Storage of Standards

9.10.1 Store unopened stock standard solutions according to the manufacturer's documented holding time and storage temperature recommendations. Protect from light.

9.10.2 Store all other working standard solutions in glass vials with Teflon lined screw caps at $\leq$ 6°C in the dark.

9.10.3 Opened stock standard solutions must be replaced after 6 months or sooner if manufacturer's expiration date comes first or comparison with quality control check samples indicates a problem.

9.10.4 All other standards must be replaced after six months or sooner if routine QC indicates a problem or manufacturer's expiration date comes first.

10.0 CALIBRATION

10.1 Initial Calibration

10.1.1 The calibration range covered for all single-component analytes employs at least five of the following standards: 1, 2, 5, 10, 25, 50, 75 and 100* μg/l (*this point may be dropped if it exceeds the linear range of the instrument). The method reporting limit is established by the concentration of the lowest standard analyzed during the initial calibration. Lower concentration standard may be needed to meet the reporting limit requirements of state specific regulatory program. The linear range covered by this calibration is the highest concentration standard. Calibration is performed for both the primary and secondary columns.

10.1.2 A calibration range must be constructed for each surrogate compound. Accordingly, add appropriate amounts of each surrogate compound to the calibration solution to define a range similar to the target compounds.

10.1.3 Unless otherwise necessary for a specific project, the analysis of the multi-component analytes (such as: Toxaphene or Chlordane) employs a single-point calibration. This single calibration standard is included with the initial calibration of the single component analytes for pattern and retention time recognition. For Ohio VAP and Dept. of Defense (DoD) projects an initial 5-point calibration is required for these analytes if there are positive hits.

10.1.4 Aliquot proper amount of each calibration standard into a 2-ml crimp top vial.

10.1.5 Prior to analysis, add 10 µl of the applicable internal standard solution (Section 9.8.1) to each standard. This results in a concentration of 50 µg/ml. Each analyte is quantitatively determined by internal standard technique.

10.1.6 For the initial calibration to be valid, the percent relative standard deviation (% RSD) (see Section 14.2) must be less than 20 % for each analyte of interest on each column. If any analyte exceeds the 20% RSD acceptance limit for a given calibration other calibration options, such as linear regression or quadratic regression not through the origin may be used or corrective action must be taken.

10.1.6.1 If the problem is associated with a standard, reanalyze the standard and recalculate the RSD.

10.1.6.2 Alternatively, the laboratory may remove individual analyte calibration levels from the lowest and/or highest levels of the curve. Multiple levels may be removed, but removal of interior levels is not permitted.

10.1.6.3 The laboratory may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. If a calibration standard was removed from the interior of the calibration, this particular standard calibration level must be removed for all analytes. Removal of calibration points from the interior of the curve is not to be used to compensate for lack of maintenance or repair to the instrument.

10.1.6.4 The laboratory must adjust the LOQ/reporting limit and quantitation range of the calibration based on the concentration of the remaining high and low calibration standards.

10.1.6.5 The laboratory must ensure that the remaining initial calibration standards are sufficient to meet the minimum requirements for number of initial calibration points as mandated by the method, or regulatory requirements.

10.1.6.6 The laboratory may replace a calibration provided that:

10.1.6.6.1 the laboratory analyzes the replacement standard within twenty-four (24) hours of the original calibration standard analysis for that particular calibration level;

10.1.6.6.2 the laboratory replaces all analytes of the replacement calibration standard if a standard within the interior of the calibration is replaced; and

10.1.6.6.3 the laboratory limits the replacement of calibration standards to one calibration standard concentration.

10.1.6.7 The laboratory must document a technically valid reason for either removal or replacement of any interior calibration point.

10.1.7 If the 20% criterion is not met for average response factor, employ a linear (5 point minimum) or quadratic (6 point minimum) model. Do not force the regression line through the origin and do not employ 0,0 as a sixth calibration standard. The correlation coefficient (r value) must be ≥0.99 for each compound to be acceptable. Additionally, the measurement of the Relative Error (%RE) must be calculated using the following equation:

$$\% \text{ Relative Error} = \frac{x'_i - x_i}{x_i} \times 100$$

x_i = true value for the calibration standard
x'_i = measured concentration of the calibration standard

This calculation shall be performed for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level. The Relative Error at both of these levels must be within 30%.

10.2 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

10.2.1 The initial calibration is verified with a second source calibration check standard from an external source (Section 9.6). At a minimum, it must be performed right after the initial calibration.

10.2.2 The percent difference (%D) (Section 14.3) for this standard must meet the %D criteria of 20% used for calibration verification on each column.

10.2.2.1 If %D is greater than 20%, reanalyze the ICV second source check or re-prepare using a fresh ampoule and reanalyze the ICV second source check standard.

10.2.2.2 If the %D criteria cannot be achieved after re-injection of the second source check standard. a new calibration curve must be prepared by making fresh calibration standards using one of the two standard sources that match each other.

10.3 Continuing Calibration Verification (CCV)

10.3.1 Continuing calibration check standards (Section 9.5.2) must be acquired at the beginning of each 12-hour shift prior to analyzing samples or after every 20 injections whichever comes first.

10.3.2 For the continuing calibration to be valid, the percent difference (%D) must be less than 20 % for each compound of interest on each column.

10.3.3 The use of an internal standard does not require all samples be bracketed with calibration verification standards. If the%D criteria fail, a continuing calibration check is allowed to be repeated only once; if the second trial fails, a new initial calibration must be performed. In situations where the first check fails to meet the criteria, the instrument logbook must have clear documented notations as to what the problem was and what corrective action was implemented to enable the second check to pass.

10.3.3.1 Retention times and area responses of internal standards must be checked in every analysis. Areas responses must be within -50% or +100% and the retention time shift cannot be > 30 seconds.

10.3.4 Ending CCVs are required for all analysis sequences. If an ending calibration verification standard fails to meet the QC criteria all samples injected after the last standard that met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results. Re-injection of the sample extracts may be required.

10.3.4.1 If the analyte was not detected in the specific samples analyzed during the analytical shift or sequence, the extracts for those samples do not need to be reanalyzed when the calibration standard response is above the initial calibration response, i.e. >20%.

Note: For DoD projects, check the bench notes for additional QC requirements.

10.3.4.2 If the analyte was detected in the specific samples analyzed during the analytical shift, or the calibration standard response is below the initial calibration response, then the extracts for those samples need to be reanalyzed.

10.3.4.3 If after rerun the ending CCV fails again, report the original run and use the second run for confirmation. Flag the compounds that were out in the first CCV in LIMS.

10.3.5 Each subsequent injection of a continuing calibration standard must be checked against the retention time windows established in Section 11.0. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is not in control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

11.0 RETENTION TIME WINDOWS

11.1 Retention time windows must be calculated for each analyte and surrogate on each GC column and whenever a new chromatographic column is installed, when a new initial calibration is analyzed or when there are significant changes in the operating conditions. The retention time windows must be reported with the analysis results in support of the identifications made.

11.2 Employ the following approach to establish retention time windows.

11.2.1 Make three injections of all single component standard mixture and multi-response products at approximately equal intervals during the 72-hr period.

11.2.2 Calculate the mean and standard deviation of the three absolute retention times-recording the retention time to three decimal places (e.g. 10.015 min) - for each single component pesticide.

11.2.3 For multi-response pesticides, choose five major peaks and calculate the mean and standard deviation of the three retention times for those peaks. The peak chosen must be fairly immune to losses due to degradation and weathering in the samples.

11.2.4 In those cases where the standard deviations of the retention time window for a particular pesticide is <0.01 minutes, the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

11.2.5 The width of the retention time window for each analyte and surrogate is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation is employed, the width of the window will be 0.03 minutes.

11.2.6 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

12.0 PROCEDURE

12.1 Sample Extraction

12.1.1 In general, water samples are extracted at a neutral pH with methylene chloride using a separate funnel (Method 3510) (Refer to SOP: EOP001 and EOP004-01). Solid samples are extracted using Method 3546 Microwave Extraction (Refer to SOP: EOP3546) or Method 3550C, Sonication (Refer to SOP: EOP003).

12.2 Sample Cleanup

12.2.1 Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. Refer to the appropriate SOPs for details.

12.2.1.1 If a sample is of biological origin, or contains high molecular weight materials, the use of Method 3640 (GPC cleanup - pesticide option) is recommended. Frequently, one of the adsorption chromatographic cleanups (alumina, silica gel, or florisil) may also be required following the GPC cleanup.

12.2.1.2 Method 3610 (alumina) may be used to remove phthalate esters.

12.2.1.3 Method 3620 (florisil) may be used to separate organochlorine pesticides from aliphatic compounds, aromatics, and nitrogen-containing compounds.

12.2.1.4 Method 3630 (silica gel) may be used to separate single component organochlorine pesticides from some interferants.

12.2.1.5 Elemental sulfur, which may be present in certain sediments and industrial wastes, interferes with the electron capture gas chromatography of certain pesticides. Sulfur must be removed by the technique described in Method 3660.

12.3 Instrument Conditions

12.3.1 Recommended instrument conditions are listed in Table 2. Modifications of parameters specified with an asterisk are allowed as long as criteria of calibration are met. Any modification must be approved by team leader/manager.

12.4 DDT and Endrin Breakdown Evaluation

12.4.1 DDT and Endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated high boiling residue from sample injection or when the injector contains metal fittings. Check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin. Presence of 4,4'-DDE, 4,4'-DDD, Endrin ketone or Endrin aldehyde indicates breakdown.

12.4.2 Before the initial calibration and at the beginning of each 12-hour shift, inject 1 μl of an evaluation standard directly on column. (Refer to Section 9.8).

12.4.3 The percent breakdown for Endrin and DDT (Section 14.7) is calculated and the breakdown report is saved in the LIMS system.

12.4.4 If degradation of either DDT or Endrin exceeds 15**%**, injector maintenance must be completed before proceeding with calibration. Refer to EQA036-01 for GC system maintenance utilized in the lab.

12.5 Initial Calibration

12.5.1 See Section 10.1.

12.6 Initial Calibration Verification (ICV)

12.6.1 Refer to Section 10.2.

12.7 Continuing Calibration Verification (CCV)

12.7.1 Refer to Section 10.3.

12.8 Sample Analysis (Primary)

12.8.1 All samples and quality control samples are injected into the Gas Chromatograph using the autosampler. Program the sampler for an appropriate number of syringe rinses and a 1ul or 2 μl injection size. A splitless injection technology is used.

12.8.2 Sample concentrations are calculated by comparing sample responses with the initial calibration of the system (Section 14.4). If sample response exceeds the limits of the

initial calibration range, dilute the extract and reanalyze. Extracts must be diluted so that all peaks are on scale, as overlapping peaks are not always evident when peaks are off scale.

12.8.3 Sample injections may continue for as long as the calibration verification standards meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

12.8.4 If chromatographic peaks are masked by the presence of interferences, further sample cleanup is necessary. Refer to Section 12.2 for extract cleanup alternatives.

12.8.4.1 If extract cleanup is required, all QC samples must also be processed through the cleanup method.

12.9 Confirmation Analysis

12.9.1 Confirmation analysis is to confirm the presence of all compounds tentatively identified in the primary analysis.

12.9.1.1 All instrument performance quality control criteria for calibration and retention times must be satisfied on the confirmation analysis.

12.9.2 Each tentative identification must be confirmed using either a second GC column of dissimilar stationary phase or using another technique such as GC/MS.

12.9.2.1 The primary and secondary analysis is conducted simultaneously in the dual-column analysis.

12.9.2.2 GC/MS confirmation may be used in conjunction with dual-column analysis if the concentration is sufficient for detection in GC/MS**,** normally a concentration of approximately 10 ng/μl in the final extract for each single component compound is required. Method 8270 is recommended as a confirmation technique when sensitivity permits.

12.9.3 Once the identification has been confirmed, the agreement between the quantitative results on both columns must be checked.

12.10 Sample Dilution

12.10.1 Establish dilution of sample in order to fall within calibration range or to minimize the matrix interference.

* Utilize screen data (specific project only).
* Utilize acquired sample data.
* Utilize the history program or approval from client/project.
* Sample characteristics (appearance).

12.10.2 If no lower dilution has been reported, the dilution factor chosen must keep the response of the largest peak for a target analyte in the upper half of the initial calibration range of the instrument.

12.10.3 Preparing Dilutions.

12.10.3.1 Prepare sample dilutions quantitatively. Dilute a stored sample extract, if available with hexane using logical volume to volume ratios, i.e., 1:5, 1:10, 1:50, etc.

12.10.3.2 Syringe dilutions. – Refer to Table 8 for dilutions. A calibrated 1ml syringe must be used to prepare dilutions. Gently shake to disperse the extract throughout the solvent prior to loading on the auto-sampler tray for further analysis.

12.10.3.3 Volumetric Flask Dilutions - Dilutions can also be made with a Class A volumetric flask. Measure the appropriate sample extract volume in a calibrated syringe and bring to final volume with dilution solvent in a Class A volumetric flask. Gently shake to disperse the extract throughout the solvent and transfer to auto-sampler vial for analysis.

12.11 Data interpretation

12.11.1 Qualitative identification

12.11.1.1 Analyst shall identify the targeted compounds with competent knowledge interpreting retention time and/or chromatographic pattern by comparison of the sample to the standard of the suspected compound. The criteria required for a positive identification are:

12.11.1.1.1 The sample component must elute at the absolute retention time window (Refer to Section 11.0) for both primary and confirmation run.

12.11.1.1.2 For the multi-response pesticides 5 major peaks are selected. The retention time window for each peak is determined from the initial calibration analysis. Identification of a multi-component analyte in the sample is based on pattern recognition in conjunction with the elution of these 5 peaks within the retention time windows of the corresponding peaks of the standard on both GC columns.

12.11.1.1.3 Be aware of matrix interfering effects on peak shape and relative peak ratios that could distort the pattern. Interpretation of these phenomena may require a highly experienced chromatographer or at least a second opinion.

12.11.2 Quantitative analysis

12.11.2.1 When a target compound has been identified, concentration (see section 14.4) will be based on the integrated area/or height of the peak and calculated by an internal standard technique. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined. Report all concentrations down to the MDL.

12.11.2.2 For multi-response pesticides, a minimum of 3 peaks are used for quantitation to calculate the calibration factors for those peaks, but 5 may be used depending on the extent of matrix interferences. These calibration factors are then used to

calculate the concentration of each corresponding peak in the sample chromatogram and the resulting concentrations are averaged to provide the final result for the sample.

12.11.2.3 When sample results are confirmed using two dissimilar columns or with two dissimilar detectors, the agreement between the quantitative results must be evaluated after the identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula in Section 14.6. Report the lower result.

12.11.2.3.1 A program to perform the RPD calculation had been developed and incorporated into ENVIROQUANT software.

12.11.2.3.2 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration.

12.11.2.3.3 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the lower result with the footnote (remark) indicating “More than 40% RPD for detected concentrations between two GC columns”.

12.11.2.3.4 If %RPD>100%, then perform additional cleanup or analyze the sample at a secondary dilution. Qualify the data as necessary, indicating potential matrix interference on the higher signal.

13.0 QUALITY CONTROL

13.1 QC Requirements Summary

DDT and Endrin Breakdown Evaluation	Every 12-hour shift
ICV -Second Source Calibration	Following initial calibration
Continuing Calibration Checks	Every 20 injections or 12 hours (whichever comes first)
Instrument Blank	After DDT/Endrin Breakdown check, and after each subsequent CCV
Method Blank	One per extraction batch* or per day for a running batch
Blank Spike	One per extraction batch* or per day for a running batch
Matrix Spike	One per extraction batch*
Matrix Spike Duplicate	One per extraction batch*
Surrogate	Every sample and standard

*The maximum number of samples per batch is twenty or per project specification.

13.2 DDT and Endrin Breakdown Evaluation

13.2.1 Refer to Section 12.4.

13.3 Initial Calibration Verification (ICV) - Second Source Calibration Check

13.3.1 Refer to Section 10.2.

13.4 Continuing Calibration Verification (CCV)

13.4.1 Refer to Section 10.3.

13.5 Instrument Blank

13.5.1 If the instrument blank contains a target analyte above its MDL, the source of the contamination must be identified and corrected before proceeding with the analysis.

13.6 Method Blank

13.6.1 The method blank is either DI water or sodium sulfate (depending upon the sample matrix) which must be extracted with each set of 20 or less samples. For a running batch, a new method blank is required for each different extraction day. The method blank are then extracted and run through any clean-up procedures along with the other samples in that batch.

13.6.2 If the method blank contains a target analyte above its MDL, the entire batch must be re-extracted and re-analyzed.

13.6.3 Surrogate compounds are added to the method blank prior to extraction and analysis. If the surrogate accuracy in the blank does not meet criteria, the entire batch must be re-extracted and re-analyzed.

13.7 Blank Spike

13.7.1 A blank spike must be extracted with each set of 20 or less samples. For a running batch, a new blank spike is required for each different extraction day. The blank spike consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. A separate blank spike may be needed if the sample requires Chlordane and/or Toxaphene. It is spiked with the same analytes at the same concentrations as the matrix spike/matrix spike duplicate.

13.7.1.1 For single-component analytes, the blank spike is prepared at 0.25 μg/l or 8.33 μg/kg on a dry weight basis.

13.7.1.2 For Toxaphene only analysis or per project specification, the blank spike is prepared at 5 μg/l or 167 μg/kg on a dry weight basis.

13.7.1.3 For Chlordane only analysis or per project specification, the blank spike is prepared at 5 μg/l or 133 μg/kg on a dry weight basis.

13.7.2 The blank spike recoveries must be assessed using in house limits.

13.7.3 If a blank spike is out of control, the following corrective actions must be taken. In the case where the blank spike recovery is high and no hits reported in associated samples and QC batch the sample results can be reported with footnote (remark) and no further action is required.

Note: For DoD projects, check the bench notes for additional QC requirements.

13.7.3.1 Check to be sure that there are no errors in the calculations, or spike solutions. If errors are found, recalculate the data accordingly.

13.7.3.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample batch.

13.7.3.3 If no problem is found, re-extract and reanalyze the sample batch.

13.8 Matrix Spike (MS)/Matrix Spike Duplicate (MSD)

13.8.1 One sample is randomly selected from each extraction batch of similar matrix types and spiked in duplicate to determine whether the sample matrix contributes bias to the analytical results.

13.8.2 A separate matrix spike and matrix spike duplicate set may be needed if the sample requires Chlordane and/or Toxaphene. Matrix spikes are prepared by spiking an actual sample for a concentration of 0.25 μg/l or 8.33 μg/kg on a dry weight basis for pesticides, 5 μg/l or 167 μg/kg for Toxaphene, 5 μg/l or 133 μg/kg for Chlordane.

13.8.3 Assess the matrix spike recoveries and relative percent difference (RPD) against the in house control limits.

13.8.4 If the matrix spike accuracy of any individual compound is out of control, the accuracy for the compound in the blank spike must be within control. Matrix interference is assumed and the data is reportable. No further corrective action is required.

13.9 Surrogates

13.9.1 Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB) are used as surrogate standards. All blanks, samples, QC samples, and calibration standards contain surrogate compounds which are used to monitor performance of the extraction, cleanup (when used), and analytical system.

13.9.2 The recoveries (refer to Section 14.5) of the surrogates must be evaluated versus the surrogate control limits developed by the laboratory.

13.9.3 If surrogate recovery is not within established control limits, corrective action must be performed if surrogate recoveries indicate that a procedural error may have occurred during the analysis of the sample.

13.9.3.1 Check the surrogate calculations for calculation or integration errors and perform corrections if detected.

13.9.3.2 Re-analyze the extract if calculation errors are not detected. If the surrogate recoveries for the re-analyzed extract are in control, report data from the re-analysis only.

13.9.3.3 If data from the reanalysis is also out of control, re-extract and reanalyze the sample.

13.9.3.4 If, upon reanalysis, the surrogate recoveries are acceptable, report the reanalysis data. If the holding time has expired prior to the reanalysis, report both the original and reanalysis results and note the holding time problem.

13.9.3.5 If the recovery is again not within limits, the problem is considered to be matrix interference. Submit both data sets with the original analysis being reported.

13.9.4 The retention time shift for surrogate must be evaluated after the analysis of each sample. The sample must be reanalyzed when the retention time of any surrogate compound is outside the retention window.

13.9.4.1 Reanalysis may not be required for samples having visible matrix interference, defined as excessive signal levels from target or non-target interfering peaks. This judgment must be approved by team leader or supervisor.

13.10 Internal Standards.

13.10.1 The use of internal standard calibration techniques does not require that all sample results be bracketed with calibration verification standards. However, when internal standard calibration is used, the retention times of the internal standards and the area responses of the internal standards must be checked for each analysis. Retention time shifts of >30 sec from the retention time of the most recent calibration standard and/or changes in internal standard areas of more than -50 to +100% are cause for concern and must be investigated.

13.11 Refer to Project Specific Bench Notes (GC8081) for additional program or client specific QC requirements.

14.0 CALCULATION

14.1 Response Factor (RF).

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:
A_s = Area of the compound being measured.
A_{is} = Area of the specific internal standard.
C_s = Concentration of the compound being measured (μg/L).
C_{is} = Concentration of the specific internal standard (μg/L).

14.2 Percent Relative Standard Deviation (% RSD).

$$\%RSD = \frac{SD}{CF_{av}} \times 100$$

Where:
SD = Standard Deviation.
CF_{av} = Average calibration factor from initial calibration.

14.3 Percent Difference (% D).

$$\% D = \frac{CF_{av} - CF_c}{CF_{av}} X\ 100$$

Where:
CF_c = CF from continuing calibration (CBCHK).

14.4 Concentration (Conc.).

14.4.1 <u>For water:</u>

$$\text{Conc. } (\mu g/l) = \frac{A_c \times M \times C_{is}}{RF_{av} \times A_{is}}$$

$$M = \frac{V_f \times D}{V_i}$$

14.4.2 <u>For soil/sediment</u> (on a dry weight basis, see EGN007):

$$\text{Conc. } (\mu g/kg) = \frac{A_c \times M \times C_{is}}{RF_{av} \times A_{is}}$$

$$M = \frac{V_f \times D}{W_s \times S}$$

Where:
A_c = Area of peak for compound being measured.
V_f = Final Volume of total extract (ml).
D = Secondary dilution factor.
V_i = Initial volume of water extracted (ml).
W_s = Weight of sample extracted (g).
S = (100 - % moisture in sample) / 100 or % solid/100.
M = Multiplier.
C_{is} = Concentration of the specific internal standard (µg/L).

14.5 Percent Recovery (% R).

$$\% R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

14.6 Relative Percent Difference (RPD).

$$RPD = \frac{|C_1 - C_2|}{(1/2)(C_1+C_2)} \times 100$$

Where:
C_1 = Matrix Spike Concentration or the result on column 1.
C_2 = Matrix Spike Duplicate Concentration or the result on column 2.

14.7 Percent Breakdown.

$$\%\ \text{Breakdown for DDT} = \frac{\text{Total DDT degradation peak area}}{\text{Total DDT peak area}} \times 100$$

Where:
Total DDT degradation peak area = DDE + DDD
Total DDT peak area = DDT + DDE + DDD

$$\%\ \text{Breakdown for Endrin} = \frac{\text{Total Endrin degradation peak area}}{\text{Total Endrin peak area}} \times 100$$

Where:
Total Endrin degradation peak area = Endrin aldehyde + Endrin ketone.
Total Endrin peak area = Endrin + Endrin aldehyde + Endrin ketone.

14.8 Linear regression by the internal standard technique.

$$C_s = \left(\frac{\frac{A_s}{A_{is}} - b}{a} \right) \times C_{is}$$

Where:
Cs = concentration of target analyte
As = Area of target analyte
Cis = concentration of the internal standard
b = Intercept
a = slope of the line

$$a = \frac{N \Sigma xy - \Sigma x \Sigma y}{N \Sigma x^2 - (\Sigma x)^2}$$

$$b = \frac{\Sigma y - a \Sigma x}{N}$$

N = number of points
x = amount of analyte
y = response of instrument

14.9 Quadratic curve with internal standard technique

$$Cs = \frac{-b \pm \sqrt{b^2 - 4a\left(c - \frac{A_s \times C_{is}}{A_{is}}\right)}}{2a}$$

Where:
Cs = concentration of target analyte
As = Area of target analyte
Cis = concentration of the internal standard
b = Intercept
a = slope of the line

14.10 Correlation Coefficient

$$r = \frac{\Sigma(x - \bar{x})(y - \bar{y})}{\sqrt{\Sigma(x - \bar{x})^2 \Sigma(y - \bar{y})^2}}$$

Where:
r = correlation coefficient
x = amount of analyte
y = response of instrument

$\bar{x}$ = average of x values

$\bar{y}$ = average of y values

15.0 DOCUMENTATION

15.1 The Analytical Logbook is a record of the analysis sequence; the logbook must be completed by the analyst daily. Each instrument will have a separate logbook. The daily sequence must be recorded in the logbook by giving a file number to every instrument standard, QC, and samples in appropriate spaces. The files must be never overwritten or skipped intentionally. In case where the file is skipped or overwritten, a thorough explanation must be documented in the notes section. Upon completion, every analytical batch must be reviewed and signed by a supervisor/team lead. Supervisor signature indicates all documentation was performed correctly.

15.1.1 If samples or blank spike require reanalysis, a brief explanation of the reason and corrective action must be documented in the Comments section. For consistency, if surrogates are high or low indicate it as (↑) for high and (↓) for low.

15.1.2 If maintenance was done on the instrument in order to pass the CCV or any other reason, the analyst must document it in the logbook.

15.2 The Standard Preparation Logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

15.2.1 The SGS Lot Number must be cross-referenced on the standard vial.

15.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

15.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

15.5 Unused blocks of any form must be x'ed or z'ed by the analyst before submitting the data for review.

15.6 Supervisory personnel must review and sign all laboratory logbooks monthly to ensure that information is being recorded properly. Additionally, the instrument maintenance logbooks and the accuracy of the recorded information must also be verified and signed off on the first page of the logbook quarterly by a supervisor/team lead.

16.0 DATA REVIEW AND REPORTING

16.1 Initial and continuing calibration check. Verify that all calibration and continuing calibration criteria have been achieved. If the criteria had not been achieved, corrective action must be performed to bring the system in control before analyzing any samples.

16.1.1 If samples had been analyzed under non-compliant calibration criteria, all sample extracts must be re-analyzed once the system is brought into control.

16.2 Quality Control Data Review. Review all QC data. If QC criteria were not achieved, perform corrective action before proceeding with analysis.

16.2.1 In some situation, corrective action may demand that the entire sample batch be re-extracted and re-analyzed before processing data.

16.3 Chromatogram Review. The chromatogram of each sample is evaluated for target compounds.

16.3.1 Check specific retention time windows for each target compound for the presence of the target compound in each chromatogram.

16.3.1.1 Each sample may require the reporting of different target compounds. Review the login to assure that the correct target compounds are identified.

16.3.2 The compound must be identified on the primary and confirmatory column before assigning a qualitative identification.

16.3.3 Manual integration of chromatographic peaks must be identified by the analysts by initialing and dating the changes made to the report.

16.4 Transfer to LIMS. Following the initial screen review, transfer the processed data to the LIMS.

17.0 POLLUTION PREVENTION & WASTE MANAGEMENT

17.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment

must be followed. All method users must be familiar with the waste management practices described in section 17.2.

17.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

17.2.1 Non hazardous aqueous wastes.
17.2.2 Hazardous aqueous wastes
17.2.3 Chlorinated organic solvents
17.2.4 Non-chlorinated organic solvents
17.2.5 Hazardous solid wastes
17.2.6 Non-hazardous solid wastes

Table 1 Target Compound List and Reporting Limits

Compound	CAS No.	Water (µg/l)	Soil (µg/kg)
alpha-BHC	319-84-6	0.01	0.67
beta-BHC	319-85-7	0.01	0.67
delta-BHC	319-86-8	0.01	0.67
Gamma-BHC (Lindane)	58-89-9	0.01	0.67
Heptachlor	76-44-8	0.01	0.67
Aldrin	309-00-2	0.01	0.67
Heptachlor epoxide	1024-57-3	0.01	0.67
Endosu1fan I	959-98-8	0.01	0.67
Dieldrin	60-57-1	0.01	0.67
4,4'-DDE	72-55-9	0.01	0.67
Endrin	72-20-8	0.01	0.67
Endosulfan II	33213-65-9	0.01	0.67
4,4'-DDD	72-54-8	0.01	0.67
Endosulfan sulfate	1031-07-8	0.01	0.67
4,4'-DDT	50-29-3	0.01	0.67
Methoxychlor	72-43-5	0.02	1.3
Endrin ketone	53494-70-5	0.01	0.67
Endrin aldehyde	7421-93-4	0.01	0.67
α-Chlordane	5103-71-9	0.01	0.67
γ-Chlordane	5103-74-2	0.01	0.67
Mirex	2385-85-5	0.02	1.3
Alachlor	15972-60-8	0.10	6.67
Hexachlorobenzene	118-74-1	0.01	0.67
2'4 DDT	789-02-6	0.01	0.67
Chlordane (technical)	12789-03-6	0.50	34
Toxaphene	8001-35-2	0.25	17

Table 2 RECOMMENDED OPERATING CONDITION	
Gas Chromatograph/Electron Capture Detectors	
Carrier Gas	Helium
Make-up gas	5 % Methane/ 95 % Argon
Make-up gas flow	*40 ml/min
Injection port temperature	*280°C
Injection type	Splitless
Detector temperature	*320°C
Column flow	2 ml/min
Gas Chromatograph Temperature Program*	
Initial temperature	*160°C
Time 1	*2 minutes
Column temperature rate 1	*45 degrees/min
Temperature 1	*200°C
Column temperature rate 2	*7 degrees/min
Temperature 2	*260°C
Column temperature rate 3	*50 degrees/min
Final temperature	*305°C
Time 3	*0.8 minutes
Total run time	10-20 minutes

*Parameter modification allowed for performance optimization as long as QC criteria are achieved.

Table 3a Pesticides and Surrogates Working Solution	
Stock Solution	**Volume Added**
Pesticides Mixture (1,000 μg/ml)	0.1 ml
Pesticides Surrogate Std Spiking Solution (200 μg/ml)	0.5ml
Mirex (1000ug/ml) (optional)	0.1ml
Hexane	9.4 ml (or 9.3 ml with Mirex)
Total	10.0 ml

Pesticides Mixture (10 μg/ml) and Surrogates (10 μg/ml) Working Solution: Prepared by measuring 0.1 ml of 1,000 μg/ml of pesticides mixture, 0.5ml of 200 μg/ml pesticides surrogate std spiking solution and bringing to 10 ml with hexane. Note larger or smaller volumes of standards may be prepared, as needed using the same ratios. ICV is prepared in the same way, but a second source is used.

Table 3b Internal Standard Working Solution	
Stock Solution	**Volume Added**
1-Bromo-2-nitrobenzene (1,000 μg/ml)	0.25 ml
Hexane	49.75 ml
Total	50.0 ml

Table 4A Pesticides Calibration Standard Solutions					
Solution	**Working Solution**	**Concentration (μg/ml)**	**Volume Added (μl)**	**Final Volume in Hexane (ml)**	**Final Concentration(μg /l)**
Standard	Pesticides Mixture	10	500	50	100

A	Surrogates	10			100
Standard	Pesticides Mixture	10	250	50	50
B	Surrogates	10			50
Standard	Pesticides Mixture	10	125	50	25
C	Surrogates	10			25
Standard	Pesticides Mixture	10	50	50	10
D	Surrogates	10			10
Standard	Pesticides Mixture	10	25	50	5
E	Surrogates	10			5
Standard	Pesticides Mixture	10	10	50	2
F	Surrogates	10			2
Standard	Pesticides Mixture	10	5	50	1
G	Surrogates	10			1
Standard	Pesticides Mixture	10	375	50	75
H	Surrogates	10			75

Standard A: Prepared by measuring 500 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard B: Prepared by measuring 250 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard C: Prepared by measuring 125 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard D: Prepared by measuring 50 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard E: Prepared by measuring 25 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard F: Prepared by measuring 10 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard G: Prepared by measuring 5 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Standard H: Prepared by measuring 375 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Table 4B Toxaphene Calibration Standard Solution (20ug/ml)	
Stock Solution	**Volume Added (µl)**
Toxaphene stock (4000 µg/ml)	125
Pesticides Surrogate Std Spiking Solution (200 µg/ml)	100
Hexane	24775
Total	25000

Toxaphene (20 μg/ml) and Surrogates (0.80 μg/l) Calibration Solution: Prepared by measuring 125 μl of 4000 μg/ml of Toxaphene stock solution, 100 μl of 200 μg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 4C Chlordane Calibration Standard Solution (20 μg/ml)	
Stock Solution	**Volume Added (μl)**
Chlordane stock (2000 μg/ml)	250
Pesticides Surrogate Std Spiking Solution (200 μg/ml)	100
Hexane	24650
Total	25000

Chlordane (20 μg/ml) and Surrogates (0.80 μg/ml) Calibration Solution: Prepared by measuring 250 μl of 2000 μg/ml of Chlordane stock solution, 100 μl of 200 μg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 4D Multi-point Toxaphene Calibration Standards (optional)					
Solution	**Stock Solution**	**Concentration (μg/ml)**	**Volume Added (μl)**	**Final Volume in Hexane (ml)**	**Final Concentration(μg/l)**
Standard A	Toxaphene	20	3750	25	3000
	Surrogate Spiking	0.8	3750		120
Standard B	Toxaphene	20	2500	25	2000
	Surrogate Spiking	0.8	2500		80
Standard C	Toxaphene	20	1250	25	1000
	Surrogate Spiking	0.8	1250		40
Standard D	Toxaphene	20	625	25	500
	Surrogate Spiking	0.8	625		20
Standard E	Toxaphene	20	312.5	25	250
	Surrogate Spiking	0.8	312.5		10
Standard F	Toxaphene	20	62.5	25	50
	Surrogate Spiking	0.8	62.5		2

Standard A: Prepared by measuring 3750 μl of 20 μg/ml of Toxaphene stock solution, 3750 μl of 0.8 μg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard B: Prepared by measuring 2500 μl of 20 μg/ml of Toxaphene stock solution, 2500 μl of 0.8 μg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard C: Prepared by measuring 1000 μl of 20 μg/ml of Toxaphene stock solution, 1000 μl of 0.8 μg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard D: Prepared by measuring 625 μl of 20 μg/ml of Toxaphene stock solution, 625 μl of 0.8 μg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard E: Prepared by measuring 312.5 μl of 20 μg/ml of Toxaphene stock solution, 312.5 μl of 0.8 μg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard F: Prepared by measuring 62.5 µl of 20 µg/ml of Toxaphene stock solution, 32.5 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 4E Multi-point Chlordane Calibration Standards (optional)					
Solution	**Stock Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration(µg/l)**
Standard A	Chlordane	20	3750	25	3000
	Surrogate Spiking	0.8	3750		120
Standard B	Chlordane	20	2500	25	2000
	Surrogate Spiking	0.8	2500		80
Standard C	Chlordane	20	1250	25	1000
	Surrogate Spiking	0.8	1250		40
Standard D	Chlordane	20	625	25	500
	Surrogate Spiking	0.8	625		20
Standard E	Chlordane	20	312.5	25	250
	Surrogate Spiking	0.8	312.5		10
Standard F	Chlordane	20	62.5	25	50
	Surrogate Spiking	0.8	62.5		2

Standard A: Prepared by measuring 3750 µl of 20 µg/ml of Chlordane stock solution, 3750 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard B: Prepared by measuring 2500 µl of 20 µg/ml of Chlordane stock solution, 2500 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard C: Prepared by measuring 1000 µl of 20 µg/ml of Chlordane stock solution, 1000 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard D: Prepared by measuring 625 µl of 20 µg/ml of Chlordane stock solution, 625 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard E: Prepared by measuring 312.5 µl of 20 µg/ml of Chlordane stock solution, 312.5 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Standard F: Prepared by measuring 62.5 µl of 20 µg/ml of Chlordane stock solution, 32.5 µl of 0.8 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 5 Continuing Calibration Check Solutions					
Checks	**Working Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration(µg/l)**
Solution 1	Pesticides Mixture	10	250	50	50
	Surrogates	10			50
Solution 2	Pesticides Mixture	10	50	50	10
	Surrogates	10			10
Solution 3	Pesticides Mixture	10	125	50	25
	Surrogates	10			25

Solution 1: Prepared by measuring 250 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Solution 2: Prepared by measuring 50 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Solution 3: Prepared by measuring 125 µl of Pesticides Mixture (10 µg/ml) and Surrogates (10 µg/ml) Working Solution and bringing to 50 ml with hexane.

Table 6 DDT and Endrin Breakdown Evaluation Standard	
Stock Solution	**Volume Added (µl)**
Pesticides Performance Evaluation Mixture (10-250 µg/ml)	50
Hexane	4995 0
Total	5000 0

DDT and Endrin Breakdown Evaluation Standard (10-250 µg/l): Prepared by measuring 50 µl of Pesticides Performance Evaluation Mixture (10-250 µg/ml) and diluting to 50 ml with hexane.

Table 7
Sample Dilution Table

All dilutions must be made using a 1ml calibrated syringe.

Dilution	*Intact Sample*	*Solvent*
1:2	500ul	500ul
1:5	200ul	800ul
1:10	100ul	900ul
1:20	50ul	950ul
1:25	40ul	960ul
1:50	20ul	980ul

Two Step dilution				
Dilution	**Step 1**		**Step 2**	
	Intact Sample	*Solvent*	*Sample Aliquot from Step 1*	*Solvent*
1:100	100ul	900ul	100ul	900ul
1:200	100ul	900ul	50ul	950ul
1:250	100ul	900ul	40ul	960ul
1:500	100ul	900ul	20ul	980ul

Three Step Dilution			
Dilution	**Step 1**	**Step 2**	**Step 3**

	Intact Sample	*Solvent*	*Sample Aliquot from Step 1*	*Solvent*	*Sample Aliquot from Step 2*	*Solvent*
1:1000	100ul	900ul	100ul	900ul	100ul	900ul
1:2000	100ul	900ul	100ul	900ul	50ul	950ul
1:2500	100ul	900ul	100ul	900ul	40ul	960ul
1:5000	100ul	900ul	100ul	900ul	20ul	980ul

Four Step Dilution

Dilution	**Step 1**		**Step 2**		**Step 3**		**Step 4**	
	Intact Sample	*Solvent*	*Sample Aliquot from Step 1*	*Solvent*	*Sample Aliquot from Step 2*	*Solvent*	*Sample Aliquot from Step 3*	*Solvent*
1:10,000	100ul	900ul	100ul	900ul	100ul	900ul	100ul	900ul
1:20,000	100ul	900ul	100ul	900ul	100ul	900ul	50ul	950ul
1:25,000	100ul	900ul	100ul	900ul	100ul	900ul	40ul	960ul
1:50,000	100ul	900ul	100ul	900ul	100ul	900ul	20ul	980ul

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
10.1.6.2	Added TNI acceptable procedure to delete points from an ICAL
4.0	MDL definition revised

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed
3.2.1; 3.2.2	MDL procedure
10.1.6.2.1	See sections 10.1.6.3-10.1.6.7

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added
10.1.6.3-10.1.6.7	TNI procedure for removing calibration points

History of Revisions

Version #	Date of Revision	Revised By
13	09/28/2018	Wahied Bayoumi
14	02/17/2020	Olga Azarian
15	03/24/2020	Maria Ruschke

END OF DOCUMENT

LAB SUPERVISOR: _____

QA OFFICER: Maria Buschke

EFFECTIVE DATE: 9-2-22

TITLE: SW846 8082A: DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs) BY GAS CHROMATOGRAPHY
REFERENCES: SW846 8082A (Revision 1, February 2007)
REVISED SECTIONS: 10.3.3

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the analytical procedures, which are utilized by SGS-Dayton to acquire samples for analysis of polychlorinated biphenyls (PCBs) as Aroclors, using dual open-tubular, capillary columns with electron capture detectors (ECD).

1.2 This gas chromatographic (GC) method applicable to the determination of the PCB Aroclors listed in Table 1 in extracts from solid and aqueous matrices.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (from 250 mL to 1 L for liquids, 15 g for solids) is extracted using the appropriate matrix-specific sample extraction technique. Petroleum Products and organic wastes are diluted with an organic solvent and follow SW 846 Method 3580A. Aqueous samples are extracted at neutral pH with methylene chloride using Method 3510 (separatory funnel). Solid samples are extracted with using Method 3546, Microwave Extraction. Wipes are extracted using Method 3550C, Sonication.

2.2 Extracts for PCB analysis may be subjected to a sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for these analytes. This cleanup technique will remove (destroy) many single components organochlorine or organophosphorus pesticides.

2.3 After cleanup, the extract is analyzed by injecting a 1 or 2-µL aliquot into a gas chromatograph with dual narrow bore fused silica capillary columns and electron capture detectors (GC/ECD). The chromatographic data may be used to determine the seven Aroclors in Table 1.

2.4 The peaks detected are qualitatively identified by comparison to retention times specific to the known target list of PCBs on two different column types (primary and confirmation).

2.5 Once identified, the Aroclor is quantitated by external standard techniques with an average calibration factor generated from a calibration curve.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve. RL's may vary depending on matrix difficulties and sample volumes or weight and percent moisture. Refer to Table 1 for current reporting limits.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B, Revision 2, and SOP EQA075.

4.0 DEFINITIONS

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. The types of blanks are Method Blank; Instrument Blank, Storage Blank, and Sulfur Blank.

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CONTINUING CALIBRATION - analytical standard run every 12 hours and at the end of analytical sequence to verify the initial calibration of the system.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the electron capture detector to the target compounds.

MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD DETECTION LIMITS (MDLs) - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is distinguishable from method blanks results. MDLs must be determined according to the procedure detailed in SOP EQA075.

PERCENT DIFFERENCE (%D) - As used in this SOP and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes

contributions from all compounds that may volatilize at or below 105° C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

REAGENT WATER - water in which an interferant is not observed at or above the minimum detection limit of the parameters of interest.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

RELATIVE RESPONSE FACTOR (RRF) - a measure of the instrument response of an analyte. Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

RETENTION TIME (RT) - the time required (in minutes) for a standard compound to elute from a chromatographic column.

SURROGATES - for semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recoveries. Surrogate are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

INSTRUMENT BLANK - a system evaluation sample containing solvent and surrogate standards added. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 Polychlorinated biphenyls have been classified as known or suspected human or mammalian carcinogens. Primary standards of these toxic compounds must be prepared in a hood. A NIOSH/Mass approved toxic gas respirator must be worn when the analyst handles high concentrations of these toxic compounds.

6.0 SAMPLE PRESERVATION AND HOLDING TIME

6.1 PRESERVATION

6.1.1 Water Samples

6.1.1.1 Samples are collected in 300mL or 1000mL glass amber bottles without preservatives.

6.1.1.2 A minimum of 250mL of an unpreserved sample is required for extraction. Additional sample volume is necessary for any samples used for matrix spike and matrix spike duplicates. Therefore, 1 liter of at least one sample in every group of 20 field samples are required for analysis to accommodate all quality control requirements.

6.1.2 Soil Samples

6.1.2.1 Samples are collected in a glass containers. No preservative is required.

6.1.3 Sample must be taken with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing possible phthalate contamination.

6.1.4 The samples must be protected from light and refrigerated at $\leq$ 6 °C from the time of receipt until extraction and analysis.

6.2 HOLDING TIME

6.2.1 Aqueous sample must be extracted within 1 year of sampling.

6.2.2 Soil sample must be extracted within 1 year of sampling.

6.2.3 Extracts must be analyzed within 40 days following extraction.

7.0 APPARATUS AND MATERIALS

7.1 GAS CHROMATOGRAPH SYSTEM

7.1.1 Gas Chromatograph-Agilent or Hewlett Packard Model 5890, 6890, and/or 7890. The analytical system comes complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for split or splitless injection with capillary columns. The capillary columns are directly coupled to the detectors.

7.1.2 Columns

7.1.2.1 Column pair 1

7.1.2.1.1 30 m x 0.32 mm fused silica (0.32 μm film thickness) ZBCLP-1 narrow-bore capillary column or equivalent.

7.1.2.1.2 30 m x 0.32 mm fused silica (0.25μm film thickness) ZBCLP-2 narrow-bore capillary column or equivalent.

7.1.2.2 Column pair 2

7.1.2.2.1 30 m x 0.32 mm fused silica (0.5 μm film thickness) RTX CLPI narrow-bore capillary column or equivalent.

7.1.2.2.2 30 m x 0.32 mm fused silica (0.25 μm film thickness) RTX CLPII narrow-bore capillary column or equivalent.

7.1.3 Detectors

7.1.3.1 Electron Capture Detectors (HP).

7.1.3.2 Micro Electron Capture Detectors (HP).

7.2 AUTOSAMPLER

7.2.1 Agilent or Hewlett Packard Model 7673A, 7683, 7643A capable of holding 100 of 2-ml crimp vials.

7.3 DATA SYSTEM

7.3.1 MSD interfaced to the gas chromatograph which allows the continuous acquisition and storage on machine readable media (disc) of all chromatographic data obtained throughout the duration of the analysis.

7.3.2 The ENVIROQUANT data system is capable of quantitation using multi-point calibration.

7.3.3 Lagato Networker with lookup database on 4mm DAT tape for long term, offline magnetic storage of data.

7.4 SYRINGE

7.4.1 Manually held ul-syringes, various volumes (Hamilton or equiv.).

7.4.2 10 μl graduated, auto sampler (Hamilton or equiv.).

8.0 REAGENTS AND STANDARDS

8.1 Refer to SGS Sample Preparation SOPs EOP001 and EOP3546 for reagents and standards used for sample extraction.

8.2 Solvents - Ultra pure, chromatography grade Hexane.

8.3 Stock standard solutions.

8.3.1 Two separate sources of commercially prepared standards with traceability documentation are used. The standards contain Aroclors 1016, 1221, 1232, 1242, 1248, 1254 and 1260. Separate calibrations are prepared and performed for Aroclors 1262 and 1268 when needed for identification of the compounds in client samples.

8.4 Working Solutions

8.4.1 Prepare working solutions, using stock solution, in hexane, as needed, that contain the compounds of interest, either singly or mixed together. Refer to Table 3A, 3B for details.

8.5 Calibration Standards

8.5.1 Initial Calibration Standards

8.5.1.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks in the other five Aroclor mixtures. As a result, a multi-point calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibration for each of the seven Aroclors. Prepare a minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260, including surrogates, by dilution of the above working solutions (Section 9.4) with hexane. Suggested levels and preparations are shown in Table 4A.

8.5.1.2 Separate calibration standards are required for the other five Aroclors. Unless otherwise necessary for a specific project, a single calibration standard near the mid-point of the expected calibration range of each remaining Aroclor is employed to determine its calibration factor and for pattern recognition. Refer to Table 4B for preparation scheme. Optional curves as shown on Table 4C may also be used for a multi-point calibration per project's specification.

8.5.2 Continuing Calibration Verification (CCV)

8.5.2.1 For Aroclor analyses, the continuing calibration checks must be a mixture of Aroclor 1016 and Aroclor 1260. Two standards at 500 μg/l and 1,000 μg/l are prepared as described in Table 5A. During the analysis, these two solutions are alternated to check the initial calibration.

8.5.2.2 In situations where only a few Aroclors are of interest for a specific project, the calibration checks of each Aroclor of interest may be prepared (Table 5B) and analyzed as the 1016/1260 mixture throughout the analytical sequence.

8.6 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

8.6.1 Prepare the ICV check standards from separate sources of stock standards from the calibration curve following the procedures in Table 6A, and 6B.

8.6.2 The ICV is prepared at 1,000 μg/l for each Aroclor and is analyzed immediately after the initial calibration.

8.7 Surrogates

8.7.1 Tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) are used as surrogate standards for this method.

8.7.2 A calibration range must be constructed for the surrogate compounds. Accordingly, appropriate amounts of surrogates are mixed with each calibration solution to define a range similar to the target compounds.

8.7.3 Surrogate compounds are also contained in continuing calibration checks, and second source calibration check standard.

8.7.4 Spike each sample, QC sample and blank with an appropriate amount of corresponding surrogate spiking solution, prior to extraction, for a final concentration in the extract of 40 µg/l of each surrogate compound.

8.8 Instrument Blank

8.8.1 An instrument blank is run after each Continuing Calibration Check. Spike hexane with an appropriate amount of surrogate spiking solution for a final concentration of 20 µg/l of each surrogate compound.

8.9 Storage of Standards

8.9.1 Store unopened stock standard solutions according to the manufacturer's documented holding time and storage temperature recommendations. Protect from light.

8.9.2 Store all other working standard solutions in glass vials with Teflon lined screw caps at ≤ 6°C in the dark.

8.9.3 Opened stock standard solutions must be replaced after 6 months or sooner if manufacturer's expiration date comes first or comparison with quality control check samples indicates a problem.

8.9.4 All other standards must be replaced after six months or sooner if routine QC indicates a problem or manufacturer's expiration date comes first.

9.0 INTERFERENCES

9.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

9.2 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned. Refer to "The Preparation of Glassware for Extraction of organic contaminants" SOP for practices utilized in the extraction department.

9.3 Interferences may be caused by contaminants that are co-extracted from the sample. The extent of the interferences will vary from source to source, which can be grouped into three broad categories.

9.3.1 Contaminated solvents, reagents, or sample processing hardware.

9.3.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

9.3.3 Compounds extracted from the sample matrix to which the detector will respond**.**

9.4 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determination.

9.4.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations. Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interference from phthalate esters.

9.4.2 Exhaustive cleanup of solvents, reagent and glassware may be required to eliminate background phthalate ester contamination.

9.4.3 These materials can be removed through the use of Method 3665 (sulfuric acid/permanganate cleanup).

9.5 Elemental sulfur is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Method 3660 is suggested for removal of sulfur.

9.6 To reduce carryover when high-concentration samples are sequentially analyzed, the syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it must be followed by the analysis of an instrument blank to check for cross contamination.

10.0 Procedure

10.1 INITIAL CALIBRATION

10.1.1 The method reporting limit is established by the concentration of the lowest standard analyzed during the initial calibration. Lower concentration standard may be needed to meet the reporting limit requirements of state specific regulatory program. The linear range covered by this calibration is the highest concentration standard.

10.1.2 The initial calibration for this method consists of two parts, described below.

10.1.2.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detectors and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. The calibration range covered for Aroclor 1016 and Aroclor 1260 employs standards of 50, 250, 500, 1,000, 2,000, and 3,000 µg/l.

10.1.2.2 Standards of the other five Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture in Section 10.1.2.1 has been used to describe the detector response. The concentration of each Aroclor standard is near the mid-point of the linear range of the detector, usually at 1,000 µg/l. The standards for these five Aroclors must be analyzed before the analysis of any samples and may be analyzed before or after the analysis of those 1016/1260 standards.

10.1.2.3 In situations where only a few Aroclors are of interest for a specific project, an initial calibration of a minimum of five standards of each Aroclors of interest instead of the 1016/1260 mixture may be performed.

10.1.3 A calibration range must be constructed for each surrogate compound. Accordingly, add appropriate amounts of each surrogate compound to the calibration solution to define a range similar to the target compounds.

10.1.4 Aliquot proper amount of each calibration standard into a 2 ml crimp top vial.

10.1.5 PCBs are quantitatively determined as Aroclors by the external standard technique. The Calibration Factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards is calculated using the equation in Section 14.1.

10.1.5.1 Use at least five peaks for the Aroclor 1016/1260 mixture, none of which are found in both of these Aroclors. At least five sets of calibration factors will be generated, each set consisting of the calibration factors for each of the five (or more) peaks chosen for this mixture.

10.1.5.2 A minimum of 3 characteristic peaks must be chosen for each of the other Aroclors, and preferably 5 peaks. The peaks must be characteristic of the Aroclor in question. Thus, each single standard will generate at least three calibration factors, one for each selected peak.

10.1.5.3 Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 6 peaks must include at least one peak that is unique to that Aroclor.

10.1.5.4 The calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. When the Aroclor 1016/1260 mixture is used to demonstrate the detector response, the calibration model chosen for this mixture must be applied to the other five Aroclors for which only single standards are analyzed. If multi-point calibration is performed for individual Aroclors, use the calibration factors from those standards to evaluate linearity.

10.1.6 For the initial calibration to be valid, the percent relative standard deviation (% RSD) (see Section 14.2) must be less than 20 % for each Aroclor of interest on each column. If any analyte exceeds the 20% acceptance limit for a given calibration, corrective action must be taken.

10.1.6.1 If the problem is associated with specific standards, reanalyze the standard and recalculate the RSD.

10.1.6.2 Alternately, the laboratory may remove individual analyte calibration levels from the lowest and/or highest levels of the curve. Multiple levels may be removed, but removal of interior levels is not permitted.

10.1.6.2.1 The laboratory may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. If a calibration standard was removed from the interior of the calibration, this particular standard calibration level must be removed for all analytes.

Removal of calibration points from the interior of the curve is not to be used to compensate for lack of maintenance or repair to the instrument.

10.1.6.2.2 The laboratory must adjust the LOQ/reporting limit and quantitation range of the calibration based on the concentration of the remaining high and low calibration standards.

10.1.6.2.3 The laboratory must ensure that the remaining initial calibration standards are sufficient to meet the minimum requirements for number of initial calibration points as mandated by the method, or regulatory requirements.

10.1.6.2.4 The laboratory may replace a calibration standard provided

10.1.6.2.4.1 the laboratory analyzes the replacement standard within twenty-four (24) hours of the original calibration standard analysis for that particular calibration level;

10.1.6.2.4.2 the laboratory replaces all analytes of the replacement calibration standard if a standard within the interior of the calibration is replaced; and

10.1.6.2.4.3 the laboratory limits the replacement of calibration standards to one calibration standard concentration.

10.1.6.2.5 The laboratory must document a technically valid reason for either removal or replacement of any interior calibration point.

10.2 INITIAL CALIBRATION VERIFICATION (ICV) - SECOND SOURCE CALIBRATION CHECK STANDARD

10.2.1 The initial calibration is verified with an ICV, a second source calibration check standard from an external source (Section 9.6). It must be performed right after the initial calibration.

10.2.2 The percent difference (%D) (Section 14.3) for this standard must meet the %D criteria of 20% used for calibration verification on each column.

10.2.2.1 If %D is greater than 20%, reanalyze the second source check. If the limit cannot be met upon re-injection, re-prepare the second source solution using a fresh ampoule and repeat the process.

10.2.2.2 If the %D criteria cannot be achieved after re-preparation of the second source, prepare a third source and repeat the process. Make fresh calibration standards using one of the two standard sources that match each other.

10.3 CONTINUING CALIBRATION VERIFICATION (CCV)

10.3.1 Continuing calibration verification (CCV) standards (Section 9.5.2) must be acquired at the beginning of each 12-hour shift, after every 10 injections not to exceed 12 hours and at the end of the analysis sequence. The 500 µg/l check standard is alternated with 1,000 µg/l standard for calibration verification.

10.3.2 For Aroclor analyses, the calibration verification standard must be a mixture of Aroclor 1016 and Aroclor 1260. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition, but the analyst may wish to include a standard for one of these Aroclors after the 1016/1260 mixture used for calibration verification throughout the analytical sequence.

10.3.3 The percent difference (%D) (see section 14.3) must be less than 20**%** for the average concentration of the characteristic peaks that make up an Aroclor of interest used for calibration verification on each column. Alternatively, if any Aroclor's single peak exceeds the 20%D acceptance criterion, this individual peak may be omitted from the average, as long as the average is calculated from a minimum of three peaks. The failing criteria Aroclor's peak that was excluded from the average must be also omitted from the analytical sequence.
The corrective action must be taken when the average for particular Aroclor exceeds 20%D.

10.3.4 Each sample analysis must be bracketed by periodic analyses of acceptable calibration verification standards every 10 injections not to exceed 12 hours. If %D criteria fails during a mid-sequence calibration check or at the end of the analysis sequence, a continuing calibration check is allowed to be repeated only once; if the second trial fails, a new initial calibration must be performed. In situations where the first check fails to meet the criteria, the instrument logbook must have clearly documented notations as to what the problem was and what corrective action was implemented to enable the second check to pass.

10.3.5 A continuing calibration standard is analyzed whenever the analyst suspects that the analytical system is out of calibration. If the calibration cannot be verified, corrective action is performed to bring the system into control. Analysis may not continue until the system is under control.

10.3.6 When a calibration verification standard fails to meet the QC criteria at the end of the analysis sequence, all samples injected after the last standard that met the QC criteria must be evaluated to prevent mis-quantitation, and re-injection of the sample extracts may be required.

10.3.6.1 If the analyte was not detected in the specific samples analyzed during the analytical shift or sequence, the extracts for those samples do not need to be reanalyzed when the calibration standard response is above the initial calibration response.

10.3.6.2 If the analyte was detected in the specific samples analyzed during the analytical shift or sequence, or the calibration standard response is below the initial calibration response, then the extracts for those samples need to be reanalyzed.

10.3.7 Each subsequent injection of a continuing calibration standard during the 12-hour analytical shift must be checked against the retention time windows established in Section 11.0. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

10.4 RETENTION TIME WINDOWS

10.4.1 Absolute retention times are used for the identification of PCBs as Aroclors. Retention time windows must be calculated for each surrogate and at least 3 to 5 characteristic peaks of each Aroclor on each GC column, when a new initial calibration is run and whenever a new chromatographic column is installed, or when there are significant changes in the operating conditions. The retention time windows must be reported with the analysis results in support of the identifications made.

10.4.2 Employ the following approach to establish retention time windows:

10.4.2.1 Make three injections of each Aroclor at approximately equal intervals during the 72-hr period.

10.4.2.2 For each Aroclor, choose three or five major peaks and calculate the mean and standard deviation of the three retention times for that peak. The peak chosen should be fairly immune to losses due to degradation and weathering in the samples. Record the retention time to three decimal places (e.g. 10.015 min) for each Aroclor.

10.4.2.3 In those cases where the standard deviations of the retention time window for a particular Aroclor is 0.01 minutes or less, the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

10.4.2.4 Apply plus or minus three times the standard deviations to retention time of each Aroclor standard (continuing calibration or middle level of initial calibration). This will be used to define the retention time window for the sample.

10.4.2.4.1 If default standard deviation of 0.01 minutes is employed, the width of the window will be 0.03 minutes.

10.4.2.5 Establish the center of the retention time window for each Aroclor and surrogate by using the absolute retention time for each Aroclor and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

10.4.2.6 When retention time windows are to be determined, analyze a standard containing DDT analogs to ensure that they do not elute at the same retention time as the last major Aroclor 1254 peak. The analyst must either adjust the GC conditions for better resolution or choose another peak that is characteristic of the Aroclor and which does not elute at the same time as of the DDT analogs.

10.5 SAMPLE EXTRACTION

10.5.1 In general, water samples are extracted at a neutral pH with methylene chloride using a separate funnel (Method 3510) (Refer to SOP: EOP001 and EOP004). Solid samples are extracted using Method 3546, Microwave Extraction (Refer to SOP: EOP3546).

10.6 SAMPLE CLEANUP

10.6.1 Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. Refer to appropriate SOPs for details.

10.6.1.1 Interferences by phthalate esters can be removed through the use of a sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for PCBs. This method must be used whenever elevated baselines or overly complex chromatograms prevent accurate quantitation of PCBs.

10.6.1.2 Element sulfur, which may be present in certain sediments and industrial wastes, interfere with the electron capture gas chromatography of certain Aroclors. Sulfur must be removed by the technique described in Method 3660.

10.7 INSTRUMENT CONDITIONS.

10.7.1 Recommended instrument conditions are listed in Table 2. Modifications of parameters specified with an asterisk are allowed as long as criteria of calibration are met. Any modification must be approved by team leader/manager.

10.8 Initial calibration

10.8.1 Refer to Section 10.1.

10.9 Initial calibration Verification (ICV) -Second source calibration check standard

10.9.1 Refer to Section 10.2.

10.10 Continuing calibration Verifications (CCV)

10.10.1 Refer to Section 10.3.

10.11 Sample analysis (Primary)

10.11.1 All samples and quality control samples are injected into the Gas Chromatograph using the autosampler. Program the sampler for an appropriate number of syringe rinses and a 1ul or 2 μl injection size. A split or splitless injection technology is used.

10.11.2 Sample concentrations are calculated by comparing sample responses with the initial calibration of the system (Section 14.4). If sample response exceeds the limits of the initial calibration range, dilute the extract and reanalyze. Extracts must be diluted so that all peaks are on scale, as overlapping peaks are not always evident when peaks are off scale.

10.11.3 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the sample meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

10.11.4 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst must consult with the source of the sample to determine whether further concentration of the sample is warranted.

10.11.5 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix.

10.12 Confirmation analysis.

10.12.1 Confirmation analysis is to confirm the presence of Aroclors tentatively identified in the primary analysis.

10.12.1.1 All instrument performance quality control criteria for calibration and retention time must be satisfied on the confirmation analysis.

10.12.2 Each tentative identification must be confirmed: using a second GC column of dissimilar stationary phase (as in the dual-column analysis), based on a clearly identifiable Aroclor pattern, or using another technique such as GC/MS.

10.12.2.1 The primary and secondary analysis is conducted simultaneously in the dual-column analysis.

10.12.2.2 GC/MS confirmation may be used in conjunction with dual-column analysis if the concentration is sufficient for detection in GC/MS, normally a concentration of approximately 10 ng/µl in the final extract for each Aroclor is required. Method 8270 is recommended as a confirmation technique when sensitivity permits.

10.12.3 Once the identification has been confirmed, the agreement between the quantitative results on both columns must be checked.

10.13 Sample Dilution

10.13.1 Establish dilution of sample in order to fall within calibration range or to minimize the matrix interference.

- Utilize screen data (specific project only).
- Utilize acquired sample data.
- Utilize the history program or approval from client/project.
- Sample characteristics (appearance, odor).

10.13.2 If no lower dilution has been reported, the dilution factor chosen must keep the response of the largest peak for a target analyte in the upper half of the initial calibration range of the instrument.

10.13.3 Preparing Dilutions.

10.13.3.1 Prepare sample dilutions quantitatively. Dilute the stored sample extract if available with hexane using logical volume to volume ratios, i.e., 1:5, 1:10, 1:50, etc.

10.13.3.2 Syringe Dilutions - A calibrated 1ml syringe must be used to prepare dilutions. Gently shake to disperse the extract throughout the solvent prior to loading on the auto-sampler tray for further analysis.

10.13.3.3 Volumetric Flask Dilutions – Dilutions can also be made with a Class A volumetric flask. Measure appropriate sample extract volume in a calibrated syringe and bring to a final volume with dilution solvent in a Class A volumetric flask. Gently shake to disperse the extract throughout the solvent prior to loading on the auto-sampler tray for further analysis.

10.14 Data interpretation

10.14.1Qualitative identification

10.14.1.1 Analyst shall identify the target analytes with competent knowledge interpreting retention time and/or chromatographic pattern by comparison of the sample to the standard of the suspected Aroclor. The criteria required for a positive identification are:

10.14.1.1.1 The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

10.14.1.1.2 The target analytes must elute within the daily absolute retention time window on both primary and confirmation column.

10.14.1.1.3 For PCBs, at least five major peaks are selected. The retention time window for each peak is determined from the initial calibration analysis. This identification of PCBs as Aroclors is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of multi-component target analytes. Tentative identification of an analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte.

10.14.1.1.4 Be aware of matrix interfering effects on peak shape and relative peak ratios which could distort the pattern. Interpretation of this phenomenon may require a highly experienced chromatographer or at least a second opinion.

10.14.2 Quantitative analysis

10.14.2.1 Once the Aroclor pattern has been identified, compare the responses of at least 3 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each corresponding peak and the linear calibration established from the multi-point calibration of the 1016/1260 mixture. A concentration (see section 14.4) based on the integrated area/or height of each of

the characteristic peaks is determined and then those resulting concentrations are averaged to provide the final result for the sample.

10.14.2.2 Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable. The quantitation may then be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs on the basis of retention times must be subtracted from the total area. When quantitation is performed in this manner, the problems must be fully described for the data user and the specific procedures employed by the analyst must be thoroughly documented.

10.14.2.3 When sample results are confirmed using two dissimilar columns or with two dissimilar detectors, the agreement between the quantitative results must be evaluated after the identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula in Section 14.6. The lower result is reported.

10.14.2.3.1 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration.

10.14.2.3.2 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the lower result with the footnote (remark) indicating “More than 40% RPD for detected concentrations between two GC columns”.

11.0 QUALITY CONTROL

11.1 QC Requirements Summary

Initial Calibration	Whenever needed
Initial Calibration Verification (ICV)	Following initial calibration
Continuing Calibration Verifications (CCV)	Every 12-hour shift, after every 10 samples and at the end of analysis sequence
Instrument Blank	After each CCV
Method blank	One per extraction batch*
Blank Spike	one per extraction batch*
Matrix Spike	one per extraction batch*
Matrix Spike Duplicate	one per extraction batch*
Surrogates	every sample and standard

*The maximum number of samples per batch is twenty or per project specification.

11.2 Initial Calibration.

11.2.1 Refer to Section 10.1.

11.3 Initial Calibration Verification (ICV) -Second Source Calibration Check.

11.3.1 Refer to Section 10.2.

11.4 Continuing Calibration Verifications (CCV)

11.4.1 Refer to Section 10.3.

11.5 Instrument Blank

11.5.1 If the instrument blank contains a target analyte above its MDL, the source of the contamination must be identified and corrected before proceeding with the analysis.

11.6 Method Blank.

11.6.1 The method blank is either DI water or ottawa sand (depending upon the sample matrix) which must be extracted with each set of 20 or less samples. For a running batch, a new method blank is required for each different extraction day. The method blank must be carried through all stages of the sample preparation and measurement.

11.6.2 If the method blank contains a target analyte above its MDL established by the laboratory, the entire batch must be re-extracted and reanalyzed.

11.6.3 Surrogate compounds are added to the method blank prior to extraction and analysis. If the surrogate accuracy in the blank does not meet criteria established by the laboratory, the entire batch must be re-extracted and reanalyzed.

11.7 Blank Spike (Laboratory Control Sample)

11.7.1 A blank spike must be extracted with each set of 20 or less samples. For a running batch, a new blank spike is required for each different day. The blank spike consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. It is spiked with the same analyte at the same concentration as matrix spike. When the presence of specific Aroclors is not anticipated, the Aroclor 1016/1260 mixture may be appropriate choice for spiking. In situations where the other Aroclors are of interest for a specific project, the analyst may employ different spiking mixtures. The blank spike is prepared at a concentration of 2 μg/l or 133.3 μg/kg (on a dry weight basis) for each Aroclor.

11.7.2 The blank spike recoveries must be assessed using in house limits established by the laboratory.

11.7.3 If a blank spike is out of control, the following corrective actions must be taken. In the case where the blank spike recovery is high, and no hits reported in associated samples and QC batch the sample results can be reported with footnote (remark) and no further action is required.

11.7.3.1 Check to be sure that there are no errors in the calculations, or spike solutions. If errors are found, recalculate the data accordingly.

11.7.3.2 Check the instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the batch.

11.7.3.3 If no problem is found, re-extract and reanalyze the batch.

11.8 Matrix Spike (MS) / Matrix Spike Duplicate (MSD).

11.8.1 One sample is randomly selected from each extraction batch and spiked in duplicate with select Aroclors to assess the performance of the method as applied to a particular matrix and to provide information on the homogeneity of the matrix. Both the MS and MSD are carried through the complete sample preparation, cleanup, and determinative procedures.

11.8.2 The MS and MSD must be spiked with the Aroclors of interest. If samples are not expected to contain target analytes, a matrix spike and matrix spike duplicate pair must be spiked with Aroclor 1016/1260 mixture. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclor must be used for spiking.

11.8.3 Matrix spikes are prepared by spiking an actual sample at a concentration 2 µg/l or 133.3µg/kg on a dry weight basis.

11.8.4 Assess the matrix spike recoveries and relative percent difference (RPD) against the control limits established by the laboratory.

11.8.5 If the matrix spike accuracy of any individual Aroclor is out of control, the accuracy for that Aroclor in the blank spike must be within control. Matrix interference is assumed, and the data is reportable. No further corrective action is required.

11.9 Surrogates.

11.9.1 Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB) are used as surrogate standards. All blanks, samples, matrix spikes, and calibration standards contain surrogate compounds which are used to monitor performance of the extraction, cleanup (when used), and analytical system.

11.9.2 The recoveries (Section 14.5) of the surrogates must be evaluated versus the surrogate control limits developed by the laboratory annually.

11.9.3 If surrogate recoveries are not within established control limits, corrective action must be performed if surrogate recoveries indicate that a procedural error may have occurred during the analysis of the sample.

11.9.3.1 Check the surrogate calculations for calculation or integration errors and perform corrections if detected.

11.9.3.2 Reanalyze the extract if no calculation errors are detected. If the surrogate recoveries for the reanalyzed extract are in control, report the data from the reanalysis only.

11.9.3.3 If the data from the reanalysis is also out of control, re-extract and reanalyze the samples

11.9.3.4 If, upon reanalysis, the surrogate recoveries are acceptable, report the reanalysis data. If the holding time has expired prior to the reanalysis, report both the original and the reanalysis results and note the holding time problem.

11.9.3.5 If recovery is again not within limits, the problem is considered to be matrix interference. Submit both data sets with the original analysis being reported.

11.9.4 The retention time shift for surrogate must be evaluated after the analysis of each sample. The sample must be reanalyzed when the retention times for both surrogates are outside the retention time window.

11.9.4.1 Reanalyses are not required for samples having visible matrix interference, defined as excessive signal levels from target or non-target interfering peaks. This judgment must be approved by a team leader or a supervisor.

11.10 Refer to Project Specific Bench Notes (GC8082) for additional program or client specific QC requirements.

11.11 Calculations

11.11.1 Calibration Factor (CF).

$$CF = \frac{A_s}{C_s}$$

where:
A_s = Area of the peak for the compound being measured.
C_s = Concentration of the compound being measured (µg/l).

11.11.2 Percent Relative Standard Deviation (% RSD).

$$\%RSD = \frac{SD}{CF_{av}} \times 100$$

where:
SD = Standard Deviation.
CF_{av} = Average calibration factor from initial calibration.

11.11.3 Percent Difference (% D).

$$\% D = \frac{|CF_{av} - CF_c|}{CF_{av}} X\ 100$$

where:
CF_c = CF from continuing calibration (CBCHK).

11.11.4 Concentration (Conc.).

For water:

$$\text{Conc. } (\mu g/l) = \frac{A_c \times M}{}$$

$$\frac{}{CF_{av}}$$

$$M = \frac{V_f \times D}{V_i}$$

For soil/sediment (on a dry weight basis, see SOP EGN007):

$$\text{Conc. } (\mu g/kg) = \frac{A_c \times M}{CF_{av}}$$

$$M = \frac{V_f \times D}{W_s \times S}$$

where:
A_c = Area of peak for compound being measured.
V_f = Final Volume of total extract (ml).
D = Secondary dilution factor.
V_i = Initial volume of water extracted (ml).
W_s = Weight of sample extracted (g).
S = (100 - % moisture in sample) / 100 or % solid/100.
M = Multiplier.

11.11.5 Percent Recovery (% R).

$$\%\ R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

11.11.6 Relative Percent Difference (RPD).

$$RPD = \frac{|C_1 - C_2|}{(1/2)(C_1 + C_2)} \times 100$$

where:
C_1 = Matrix Spike Concentration or the result on column 1.
C_2 = Matrix Spike Duplicate Concentration or the result on column 2.

12.0 DOCUMENTATION

12.1 The Analytical Logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

12.1.2 If samples require reanalysis, a brief explanation of the reason must be documented in this log. For consistency, if surrogates are high or low indicate it as (↑) for high and (↓) for low.

12.2 The Standard Preparation Logbook must be completed for all standard preparations. All information requested must be completed, the page must be signed and dated by the respective person.

12.2.2 The SGS Lot Number must be cross-reference on the standard vial.

12.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

12.5 Unused blocks of any form must be X'ed and Z'ed by the analyst before submitting the data for review.

12.6 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information must also be verified during this review.

13.0 DATA REVIEW AND REPORTING

13.1 Initial and continuing calibration check. Verify that all calibration and continuing calibration criteria have been achieved. If the criteria had not been achieved, corrective action must be performed to bring the system in control before analyzing any samples.

13.1.1 If samples had been analyzed under non-compliant calibration criteria, all sample extracts must be re-analyzed once the system is brought into control.

13.2 Quality Control Data Review. Review all QC data. If QC criteria were not achieved, perform corrective action before proceeding with analysis.

13.2.1 In some situation, corrective action may demand that the entire sample batch be re-extracted and re-analyzed before processing data.

13.3 Chromatogram Review. The chromatogram of each sample is evaluated for target analytes.

13.3.1 Check specific retention time windows for each target compound for the presence of the target compound in each chromatogram.

13.3.1.1 Each sample may require the reporting of different target analytes. Review the login to assure that the correct target compounds are identified.

13.3.2 The Aroclor must be identified on the primary and confirmatory column before assigning a qualitative identification.

13.3.3 Manual integration of chromatographic peaks must be identified by the analysts. An electronic signature is applied upon data review.

13.4 Transfer to LIMS. Following the initial screen review, transfer the processed data to the LIMS.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 17.2.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non-hazardous aqueous wastes.

14.2.2 Hazardous aqueous wastes

14.2.3 Chlorinated organic solvents

14.2.4 Non-chlorinated organic solvents

14.2.5 Hazardous solid wastes

14.2.6 Non-hazardous solid wastes

Table 1. PCB Aroclors and Reporting Limits				
Compound	**CAS Number**	**Water (µg/l)**	**Soil (µg/kg)**	**Oil (µg/kg)**
Arochlor – 1016	12674-11-2	0.5	33.5	500
Arochlor – 1221	11104-28-2	0.5	33.5	500
Arochlor – 1232	11141-16-5	0.5	33.5	500
Arochlor – 1242	53469-21-9	0.5	33.5	500
Arochlor – 1248	12672-29-6	0.5	33.5	500
Arochlor – 1254	11097-69-1	0.5	33.5	500
Arochlor – 1260	11096-82-5	0.5	33.5	500
Arochlor – 1262	37324-23-5	0.5	33.5	500
Arochlor – 1268	11100-14-4	0.5	33.5	500

Table 2. RECOMMENDED OPERATING CONDITION	
Gas Chromatograph/Electron Capture Detectors	
Carrier Gas	Helium
Make-up gas	5 % Methane/ 95 % Argon
Make-up gas flow	*30 ml/min
Injection port temperature	*****235 °C
Injection type	Split / Splitless
Detector temperature	*****320 °C
Column flow	*5 ml/min
Gas Chromatograph Temperature Program*	
Initial temperature	*170 °C
Time 1	*2 min
Column temperature rate 1	*30 degrees/min
Temperature 1	*180 °C
Column temperature rate 2	*3.5 degrees/min
Temperature 2	*240 °C
Column temperature rate 3	*10 degrees/min
Final temperature	*280 °C
Time 3	*5 min
Total run time	30-40 min

* Parameter modification allowed for performance optimization as long as QC criteria are achieved.

Table 3A. Aroclors 1016/1260 Mixture and Surrogates Working Solution	
Stock Solution	**Volume Added**
Aroclor 1016/1260 (1,000 µg/ml)	500 µl
Pesticides Surrogate Std Spiking Solution (200 µg/ml)	100 µl
Hexane	fill to volume
Total	25.0 ml

- Aroclors 1016/1260 (20 µg/ml) and Surrogates (0.8 µg/ml) Working Solution: Prepared by measuring 500 µl of 1,000 µg/ml Aroclor 101/1260 and 100µ of 200 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 3B. Individual Aroclor* and Surrogates Working Solution	
Stock Solution	**Volume Added**
Individual Aroclor* (1,000 µg/ml)	500 µl
Pesticides Surrogate Std Spiking Solution (200 µg/ml)	100 µl
Hexane	24.4 ml
Total	25 ml

*Aroclor: 1221, 1232, 1242, 1248, 1254, 1262 & 1268

- Individual Aroclor (20 µg/ml) and Surrogates (0.8 µg/ml) Working Solution: Prepared by measuring 500 µl of 1,000 µg/ml each individual Aroclor, 100 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 4A. Aroclors 1016/1260 Calibration Standard Solutions					
Standard	**Working Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration (µg/l)**
Standard A	Aroclors 1016/1260	20	62.5	25	50
	Surrogates	0.8			2
Standard B	Aroclors 1016/1260	20	312.5	25	250
	Surrogates	0.8			10
Standard C	Aroclors 1016/1260	20	625	25	500
	Surrogates	0.8			20
Standard D	Aroclors 1016/1260	20	1250	25	1,000
	Surrogates	0.8			40
Standard E	Aroclors 1016/1260	20	2,500	25	2,000
	Surrogates	0.8			80
Standard F	Aroclors 1016/1260	20	3,750	25	3,000
	Surrogates	0.8			120

- Standard A: Prepared by measuring 62.5 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A) and bringing to 25 ml with hexane.

- Standard B: Prepared by measuring 312.5 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A) and bringing to 25 ml with hexane.

- Standard C: Prepared by measuring 625 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A) and bringing to 25 ml with hexane.

- Standard D: Prepared by measuring 1,250 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A) and bringing to 25 ml with hexane.

- Standard E: Prepared by measuring 2,500 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A) and bringing to 25 ml with hexane.

- Standard F: Prepared by measuring 3,750 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A) and bringing to 25 ml with hexane.

Table 4B. Single-Point Calibration Standard (1,000 µg/l) for Individual Aroclor*	
Stock Solution	**Volume Added**
Individual Aroclor*/Surrogate Working Solution (20 µg/ml/0.80µg/ml) (Table 3B)	1,250 µl
Hexane	23.75 ml
Total	25 ml

* Aroclor: 1221, 1232, 1242, 1248, 1254, 1262, & 1268.

- Individual Aroclor Calibration Standard (1,000 µg/l) and Surrogates (40 µg/l) Solution: Prepared by measuring 1,250 µl of individual Aroclor and surrogates working solution, containing 20 µg/ml of each corresponding Aroclor and 0.80 µg/ml of both surrogate compounds, and bringing to 25 ml with hexane.

Table 4C. Multi-point Calibration Standards for Individual Aroclor* (optional)					
Standard	**Stock Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration(µg/l)**
Standard A	Aroclor*	20	62.5	25	50
	Surrogates	0.8			2
Standard B	Aroclor*	20	312.5	25	250
	Surrogates	0.8			10
Standard C	Aroclor*	20	625	25	500
	Surrogates	0.8			20
Standard D	Aroclor*	20	1250	25	1,000
	Surrogates	0.8			40
Standard E	Aroclor*	20	2,500	25	2,000
	Surrogates	0.8			80
Standard F	Aroclor*	20	3,750	25	3,000
	Surrogates	0.8			120

*Aroclor: 1221, 1232, 1242, 1248, 1254, 1262, & 1268.

- Standard A: Prepared by measuring 62.5 µl of Individual Aroclor and Surrogates Working Solution (Table 3B) and bringing to 25 ml with hexane.

- Standard B: Prepared by measuring 312.5 µl of Individual Aroclor and Surrogates Working Solution (Table 3B) and bringing to 25 ml with hexane.

- Standard C: Prepared by measuring 625 µl of Individual Aroclor and Surrogates Working Solution (Table 3B) and bringing to 25 ml with hexane.

- Standard D: Prepared by measuring 1,250 µl of Individual Aroclor and Surrogates Working Solution (Table 3B) and bringing to 25 ml with hexane.

- Standard E: Prepared by measuring 2,500 µl of Individual Aroclor and Surrogates Working Solution (Table 3B) and bringing to 25 ml with hexane.

- Standard F: Prepared by measuring 3,750 µl of Individual Aroclor and Surrogates Working Solution (Table 3B) and bringing to 25 ml with hexane.

Table 5A. Continuing Calibration Check Solutions for Aroclors 1016/1260					
Checks	**Working Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration (µg/l)**
Solution 1	Aroclors 1016/1260	20	625	25	500
	Surrogates	0.8			20
Solution 2	Aroclors 1016/1260	20	1250	25	1,000
	Surrogates	0.8			40

- Solution 1: Prepared by measuring 625 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A) and bringing to 25 ml with hexane.

- Solution 2: Prepared by measuring 1,250 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A) and bringing to 25 ml with hexane.

Table 5B. Continuing Calibration Check Solutions for Individual Aroclor*					
Checks	**Working Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration (µg/l)**
Solution 1	Aroclor*	20	625	25	500
	Surrogates	0.8			20
Solution 2	Aroclor*	20	1250	25	1,000
	Surrogates	0.8			40

* Aroclor: 1221, 1232, 1242, 1248, 12541262, & 1268

- Solution 1: Prepared by measuring 625 µl of Individual Aroclor and Surrogates Working Solution (Table 3B) and bringing to 25 ml with hexane.

- Solution 2: Prepared by measuring 1,250 µl of Individual Aroclor and Surrogates Working Solution (Table 3B) and bringing to 25 ml with hexane.

Table 6A. Second Source Calibration Check Standard for Aroclors 1016/1260 (1,000 µg/l)	
Stock Solution	**Volume Added**
Aroclors 1016/1260 (25 µg/ml) and Surrogates (2.5 µg/ml) Working Solution	1,000 µl
Hexane	24 ml
Total	25 ml

- Aroclors 1016/1260 (25 µg/ml) and Surrogates (2.5 µg/ml) Working Solution: Prepared by measuring 250 µl of 1,000 µg/ml Aroclors 1016/1260 mix solution (2nd source), 125 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 10 ml with hexane.

- Aroclors 1016/1260 (1,000 µg/l) and Surrogates (100 µg/l) Solution: Prepared by measuring 1,000 µl of Aroclors 1016/1260 (25 µg/ml) and surrogates (2.5 µg/ml) working solution and bringing to 25 ml with hexane.

Table 6B. Second Source Calibration Check Standard for Individual Aroclor* (1,000 μg/l)	
Stock Solution	**Volume Added**
Individual Aroclor* (100μg/ml)	250uL
Surrogates (200 μg/ml)	5uL
Hexane	Add to bring to 25mL volume
Total	25 ml

*Aroclor: 1221, 1232, 1242, 1248, 1254, 1262 & 1268

- ICV preparation: Using Individual Aroclor (100 μg/ml) and Surrogates (200 μg/ml) Stock Solutions : Measure 250 μl of 1,00 μg/ml each individual Aroclor stock solution 2nd source and 5μl of 200 μg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
10.3.3	Added acceptance criteria for CCV

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
09	10/04/2018	Wahied Bayoumi
10	10/27/2020	Wahied Bayoumi
11	11/06/2020	Olga Azarian
12	03/12/2021	Olga Azarian
13	9/1/2022	Jason Savoie

END OF DOCUMENT

LAB SUPERVISOR: _____

QA OFFICER: _____

EFFECTIVE DATE: 4-21-2020

TITLE: CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION
REFERENCES: SW846 8151A (Revision 1, December 1996)
REVISED SECTIONS: 3.2, 4.0 (MDL),10.2.6
DELETED SECTIONS: 3.2.1, 3.2.2

1.0 Scope & Application

1.1 Method 8151A is a capillary gas chromatographic (GC) method for determining certain chlorinated acid herbicides and related compounds in aqueous, soil and waste matrices. Specifically, Method 8151A may be used to determine the following compounds:

Compound	CAS Number [a]
2,4-D	94-75-7
2,4-DB	94-82-6
2,4,5-TP (Silvex)	93-72-1
2,4,5-T	93-76-5
Dalapon	75-99-0
Dicamba	1918-00-9
Dichloroprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2
Pentachlorophenol	87-86-5
Picloram	1918-02-1

[a] Chemical Abstract Service Registry Number

1.2 Because these compounds are produced and used in various forms (i.e., acid, salt, ester, etc.), Method 8151A describes a hydrolysis step that can be used to convert herbicide esters into the acid form prior to analysis.

2.0 Summary

2.1 Method 8151A is a procedure for extracting, derivatizing, and analyzing sample extracts by gas chromatography for chlorinated acid herbicides in water, soil, and waste samples. It also includes procedures for the hydrolysis and analysis of herbicide esters.

2.2 Water samples are extracted with diethyl ether and then esterified with diazomethane. The

derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.3 Soil and waste samples are extracted and esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.4 If herbicide esters are to be determined using this method, hydrolysis conditions for the esters in water and soil extracts are described.

3.0 Reporting Limit & Method Detection Limit

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve. This concentration is defined as the lower limit for quantitative accuracy. The reporting limits for the herbicides that can be determined by this method range from 5 μg/l to 5000 μg/l (compound dependent). The reporting limits for a specific waste sample may vary depending upon the sample size, moisture content, nature of interferences present, and the sample matrix. (See Table 6).

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B, Revision 2. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

4.0 Definitions

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank, Instrument Blank, Storage Blank, and Sulfur Blank**.**

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

CONTINUING CALIBRATION - analytical standard run every 12 hours to verify the initial calibration of the system.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the electron capture detector to the target compounds.

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards, and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

METHOD DETECTION LIMITS (MDLs) - The minimum concentration of a substance that can be measured and reported with 99% confidence that the measured concentration is distinguishable from method blank results.

REAGENT WATER - water in which an interferant is not observed at or above the minimum detection limit of the parameters of interest.

RESOLUTION - also termed separation or percent resolution, the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

RETENTION TIME (RT) - the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

SURROGATES (Surrogate Standard) - for semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

INSTRUMENT BLANK – a system evaluation sample containing hexane surrogate standards added. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

5.0 Health & Safety

5.1 The analyst must follow normal safety procedures as outlined in the SGS Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 Several analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens. Primary standards of target analytes must be prepared in a hood. A NIOSH/MESA approved toxic gas respirator must be worn when the analyst handles high concentrations of these toxic compounds.

5.4 Diazomethane must be used only by experienced analysts, due to the potential hazards associated with its use.

6.0 Collection, Preservation, & Holding Times

6.1 Water samples may be collected in 1 L (or 1 quart) amber glass containers, fitted with screw-caps lined with Teflon. If amber containers are not available, the samples should be protected from light. Soil samples may be collected in glass containers or closed end tubes (e.g., brass sleeves) in sufficient quantity to perform the analysis.

6.2 All samples must be iced or refrigerated at 0 - 6°C from the time of collection until extraction.

6.3 Procedure for Sample Storage

6.3.1 The samples must be protected from light and refrigerated at 0 - 6°C.

6.3.2 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants.

6.4 Procedure for Sample Extract Storage

6.4.1 Sample extracts must be protected from light and stored at 0 - 6°C. Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants. Samples, sample extracts, and standards must be stored separately.

6.5 Holding Times

6.5.1 Extraction of water samples must be completed within seven (7) days of sample collection. Extraction of soil/sediment samples must be completed within fourteen (14) days of sample collection.

6.5.2 Analysis of sample extracts must be completed within 40 days following the start of extraction.

7.0 Apparatus and Materials

7.1 Gas Chromatographic System

7.1.1 Gas Chromatograph. HP-5890 or HP-6890 Model which includes an analytical system completes with a temperature programmable gas chromatograph. Accessories required include syringes, capillary chromatographic columns, and gases. The capillary column is directly coupled to the source. The chromatograph has a Grob type injector.

7.1.2 GC columns. Narrow-bore columns – two required:

7.1.2.1 Column 1 - 30 m x 0.32 mm, (RTX CLP 1, DB-5, J&W Scientific, or equivalent),

7.1.2.2 Column 2 - 30 m x 0.32 mm, (RTX-CLP2, DB-1701, J&W Scientific, or equivalent),

7.1.3 Electron Capture Detectors (ECD).

7.2 Data System

7.2.1 HP Chemstation interfaced to the gas chromatograph which allows the continuous acquisition and storage on machine-readable media (disc) of all chromatographic data obtained throughout the duration of the analysis.

7.2.2 The ENVIROQUANT data system is capable of quantitation using multi-point calibration.

7.2.3 Lagato Networker with lookup database on 4mm DAT tape for long term, off line magnetic storage of data.

7.3 Autosampler

7.3.1 Hewlett Packard Model 7673A / 7683, capable of holding 100 of 2-ml vials.

7.4 Microsyringe - 10-uL.

8.0 Standards & Reagents

8.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 Solvents - All solvents should be pesticide quality or equivalent.

8.3 Stock Standard Solutions.

8.3.1 Certified, commercially prepared standards, from two separate sources are used. One source is used for the initial calibration and a second source is used for the calibration verification.

The stock standard solutions can be stored at 0-6°C, protected from light until the manufacturer's expiration date.

8.4 Intermediate Calibration Standard Solution.

8.4.1 The intermediate solution is prepared by adding an appropriate amount of each stock standard and surrogate compound into a 10 ml volumetric flask. Dilute the solution to the volume with hexane and mix thoroughly. The final concentrations range from 200 μg/l – 100 mg/l depending upon the compounds. Refer to Table 2 for details.

8.4.2 Store this solution in a refrigerator at 0-6°C for six months or until the manufacturer's expiration date, whichever is sooner.

8.5 Calibration Standards.

8.5.1 Initial Calibration Standards

8.5.1.1 Calibration standards should be made by quantitative dilutions of the above intermediate solution. Suggested levels and preparations are shown in Table 3A and 3B. All standards should be brought to the final volume with hexane.

8.5.1.2 A calibration range must be constructed for each surrogate compound. Accordingly, add appropriate amounts of each surrogate compound to the calibration solution to define a range similar to the target compounds (Table 3B).

8.5.2 Continuing Calibration Verification.

8.5.2.1 Two standards near the mid-point of the calibration range are prepared as described in Table 3A Standards 3 and 4. During the analysis, these two solutions are run alternatively to check the initial calibration.

8.5.3 Calibration solutions must be replaced after six months, or before manufacturer's expiration date or sooner if comparison with check standards indicates a problem.

8.6 Initial Calibration Verification (ICV) -Second Source Calibration Check Standard.

8.6.1 Prepare an intermediate solution of the herbicide mixture from an alternative supplier per Section 8.10. If an alternate source cannot be identified, obtain a stock solution from the same vendor that is from a different stock as indicated by the stock number.

8.6.2 Prepare a second source calibration check standard following Table 5 by using the above intermediate solution.

8.7 Surrogate standards - Spike each sample, standard, and blank with the surrogate solution, 2,4-Dichlorophenylacetic acid (DCAA), to encompass the range of the temperature program used in this method.

8.7.1 The surrogate solutions from two different sources are prepared from commercially purchased standards. One source is used for the initial calibration and a second source is used for second source calibration check standard.

8.7.2 Prepare the surrogate standard by diluting 2 ml of 5000 ppm stock standard to 1 liter in acetone for a concentration of 10 ppm.

8.7.3 Add 0.5 ml of the surrogate spiking solution to samples blanks and QC samples, prior to extraction, resulting in a final concentration in the extract of 0.5mg/L or 1.0 mg/kg.

9.0 Interferences

9.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks.

9.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from waste to waste, depending upon the nature and diversity of the waste being sampled.

9.3 Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

9.4 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids.

9.5 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

9.6 Sample extracts should be dry prior to methylation or else poor recoveries will be obtained.

10.0 Procedure

10.1 Refer to EOP8151 for extraction procedures.

10.2 INITIAL CALIBRATION.

10.2.1 The calibration range covered for this analysis is employs standards in Table 3B. The method reporting limit is established by the concentration of the lowest standard analyzed during the initial calibration. Lower concentration standard may be needed to meet the reporting limit requirements of state specific regulatory program. The linear range covered by this calibration is the highest concentration standard. A minimum of 5 standards must be used for the initial calibration.

10.2.2 A calibration range must be constructed for the surrogate compound. Accordingly, add appropriate amounts of 2,4-Dichlorophenylacetic acid (DCAA) to the calibration solution to define a range similar to the target compounds.

10.2.3 Program the GC system and autosampler to the GC conditions detailed in Table 1. Load the calibration standards into the auto sampler for analysis and initiate the calibration run.

10.2.4 Each analyte is quantitatively determined using the external standard technique. The calibration factor (CF) is defined in Section 15.1.

10.2.5 For the initial calibration to be valid, the RSD of the mean calibration factors must be less than or equal to 20% over the calibration range on each column. If any analyte exceeds the 20% acceptance limit for a given calibration, corrective action must be taken.

10.2.6 If the problem is associated with a single standard, the lab may remove and replace calibration standards if the following requirements are met:

10.2.6.1 The laboratory may remove individual analyte calibration levels from the lowest and/or highest levels of the curve. Multiple levels may be removed, but removal of interior levels is not permitted.

10.2.6.2 The laboratory may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. If a calibration standard was removed from the interior of the calibration, this particular standard calibration level must be removed for all analytes. Removal of calibration points from the interior of the curve is not to be used to compensate for lack of maintenance or repair to the instrument.

10.2.6.3 The laboratory must adjust the LOQ/reporting limit and quantitation range of the calibration based on the concentration of the remaining high and low calibration standards.

10.2.6.4 The laboratory must ensure that the remaining initial calibration standards are sufficient to meet the minimum requirements for number of initial calibration points as mandated by the method, or regulatory requirements.

10.2.6.5 The laboratory may replace a calibration standard provided that:

10.2.6.5.1 the laboratory analyzes the replacement standard within twenty-four (24) hours of the original calibration standard analysis for that particular calibration level;

10.2.6.5.2 the laboratory replaces all analytes of the replacement calibration standard if astandard within the interior of the calibration is replaced; and

10.2.6.5.3 the laboratory limits the replacement of calibration standards to one calibration standard concentration

10.2.6.6 The laboratory must document a technically valid reason for either removal or replacement of any interior calibration point.

10.3 INITIAL CALIBRATION VERIFICATION (ICV) - SECOND SOURCE CALIBRATION CHECK

10.3.1 The calibration is verified with a second source calibration check standard (ICV) at mid-level concentration from an independent source (Section 8.12). It must be performed right after the initial calibration.

10.3.2 The percent difference (% D) (Section 15.3) for all the target compounds must meet the %D criteria of 20% used for calibration verification on each column.

10.3.2.1 If % D is greater than 20%, reanalyze the second source check. If the criteria cannot be met upon re-injection, re-prepare the second source solution using a fresh ampoule and repeat the process.

10.3.2.2 If the %D criteria cannot be achieved after re-preparation of the second source, prepare a third source and repeat the process. Make fresh calibration standards using one of the two standard sources that match each other.

10.4 CONTINUING CALIBRATION VERIFICATIONS (CCV)

10.4.1 Continuing calibration verification standards (Section 8.11.2) must be acquired after every 10 injections or every 12 hours, whichever is more frequent, and at the end of the analysis sequence. Two different level standards are alternated to serve as a further check on the initial calibration.

10.4.2 This check standard must be within 20% of the calibration response factor from the initial calibration on each column. Calculate the percent difference for calibration verification of linear calibrations by evaluating the instrument response difference between the initial calibration and each subsequent analysis of the verification standard. Calculate the percent difference (%D) using the following formula in Section 15.3.

10.4.3 Each sample analysis must be bracketed with acceptable calibration verification standards, run after every 10 injections or 12-hours whichever is more frequent. More frequent analyses of standards will minimize the number of sample extracts that would have to be re-injected if the QC limits are violated. If %D criteria fails during a mid sequence calibration check or at the end of the analysis sequence, a continuing calibration check is allowed to be repeated only once; if the second trial fails, a new initial calibration must be performed and the samples may need to be reanalyzed. In situations where the first check fails to meet the criteria, the instrument logbook should have clear documented notations as to what the problem was and what corrective action was implemented to enable the second check to pass.

10.4.4 When a calibration verification standard fails to meet the QC criteria at the end of the analysis sequence, all samples injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations, and re-injection of the sample extracts may be required.

10.4.4.1 The analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed when the calibration standard response is above the initial calibration response.

10.4.4.2 The analyte was detected in the specific samples analyzed during the analytical shift, or the calibration standard response is below the initial calibration response, then the extracts for those samples need to be reanalyzed.

10.4.5 Each subsequent injection of a continuing calibration standard during the 12-hour analytical shift must be checked against the retention time windows established in Section 12.0. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

10.5 RETENTION TIME WINDOWS

10.5.1 The absolute retention time windows must be calculated for each analyte and surrogate on each GC column and instrument. New retention time windows must be established after each Initial Calibration, whenever a new chromatographic column is installed, or when there are significant changes in the operating conditions. The retention time windows must be reported with the analysis results in support of the identifications made.

10.5.2 Employ the following approach to establish retention time windows.

10.5.2.2 Make three injections of all standard mixtures over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

10.5.2.3 Record the retention time for each analyte and surrogate to three decimal places (e.g., 0.007). Calculate the mean and standard deviation of the three absolute retention times for each analyte and surrogate

10.5.2.4 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes

10.5.2.5 The width of the retention time window for each analyte and surrogate is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation is employed, the width of the window will be 0.03 minutes

10.5.2.6 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration

10.5.3 The surrogate retention times in the calibration standards are useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time window, the analyst must determine the cause and correct the problem before continuing analyses.

10.6 GAS CHROMATOGRAPH CONDITIONS.

10.6.1 Establish instrument conditions following the specifications detailed in Table 1. Modifications of parameters specified with an asterisk are allowed as long as criteria of calibration are met. Any modification should be approved by team leader/manager.

10.7 SAMPLE EXTRACTS ANALYSIS (PRIMARY).

10.7.1 All samples and quality control samples are injected into the Gas Chromatograph using the autosampler. Program the sampler for an appropriate number of syringe rinses and a 1ul or 2ul injection size. A splitless injection technology is used.

10.7.2 Sample concentrations are calculated by comparing sample responses with the initial calibration of the system (Section 15.4). If sample response exceeds the limits of the initial calibration range, dilute the extract and reanalyze. Extracts should be diluted so that all peaks are on scale, as overlapping peaks are not always evident when peaks are off scale.

10.7.3 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the sample meet instrument QC requirements. The sequence ends when the last set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

10.7.4 If chromatographic peaks are masked by the presence of interferences, further sample cleanup is necessary. Refer to the sample preparation steps for extract cleanup alternatives.

10.7.4.1 If extract cleanup is required, all QC samples must also be processed through the cleanup method.

10.8 CONFIRMATION ANALYSIS.

10.8.1 Confirmation analysis is to confirm the presence of all compounds tentatively identified in the primary analysis. The primary and confirmation (secondary) analysis is conducted simultaneously.

10.8.1.1 All instrument performance quality control criteria for calibration and retention times must be satisfied on the confirmation column.

10.8.2 Each tentative identification must be confirmed using either a second GC column of dissimilar stationary phase or using anther technique such as GC/MS.

10.8.2.1 GC/MS confirmation may be used in conjunction with dual-column analysis if the concentration is sufficient for detection in GC/MS. Refer to Standard Operating Procedure for Method SW846 8270 for the appropriate GC/MS operating conditions and analysis procedures.

10.8.2.1.1 GC/MS will normally require a concentration of approximately 10ng/µl in the final extract for each compound.

10.9 SAMPLE DILUTION.

10.9.1 Establish dilution of sample in order to fall within calibration range or to minimize the matrix interference.

- Utilize screen data (specific project only).
- Utilize acquired sample data.
- Utilize the history program or approval from client/project.
- Sample characteristics (appearance, odor).

10.9.2 If no more lower dilution been reported, the dilution factor chosen should keep the response of the largest peak for a target compound in the upper half of the initial calibration range of the instrument.

10.9.3 Preparing Dilutions.

10.9.3.1 Prepare sample dilutions quantitatively. Dilute the sample extract with hexane using logical volume to volume ratios, i.e., 1:5, 1:10, 1:50, etc.

10.9.3.2 Transfer an appropriate amount of extract to a volumetric flask. Fill the flask to the volume mark with hexane. Gently shake to disperse the extract throughout the solvent. Alternately, a syringe may be used to prepare dilutions

10.10 DATA INTERPRETATION.

10.10.1 Qualitative identification.

10.10.1.1 The compounds shall be identified by analysts with competent knowledge interpreting retention time and/or chromatographic pattern by comparison of the sample to the standard of the suspected compound. The criteria required for a positive identification are:

10.10.1.1.1 The sample component must elute at the daily absolute retention time window for both primary and confirmation column.

10.10.1.1.2 Be aware of matrix interfering effects on peak shape and relative peak ratios which could distort the pattern. Interpretation of this phenomenon may require a highly experienced analyst or minimally a second opinion.

10.10.2 Quantitative analysis.

10.10.2.1 When a target compound has been identified, concentration (see section 15.4) will be using the integrated area of the sample peak or, as an alternative, the sample peak height and calculated by external standard technique.

10.10.2.1.1 If the calibration standards have been analyzed in the same manner as the samples, then no correction factor is required. However if the calibration is performed using standards made from methyl ester compounds (compounds not esterified by application of this method), then the calculation of the concentration must include a correction for the molecular weight of the methyl ester versus the acid herbicide (see table 7).

10.10.2.2 When sample results are confirmed using two dissimilar columns or with two dissimilar detectors, the agreement between the quantitative results must be evaluated after the identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula in Section 15.6. The lower result is reported.

10.10.2.2.1 A program to perform the RPD calculation had been developed and incorporated into ENVIROQUANT software. Whenever an analyte has a RPD exceeds 40% between the results on the primary and secondary columns, a “P” qualifier or asterisk will be shown on the quantitation report next to its results.

10.10.2.2.2 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the

baseline parameters established by the instrument data system (or operator) during peak integration.

10.10.2.2.3 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the lower result with the footnote on the LIMS result page indicating "More than 40% RPD for detected concentrations between two GC columns".

11.0 Quality Assurance

11.1 Control parameters have been established for critical steps within this method that enable the analyst to assess method performance and conduct corrective action for defective processes before, during and after the analysis of samples. The analyst must exercise his/her responsibilities to take corrective action if any of the following controls are not within established specifications.

Initial Calibration	Whenever needed
Initial Calibration Verification (ICV) -Second Source	Following initial calibration
Continuing Calibration Verifications (CCV)	Every 12 hours or 10 samples whichever is more frequent.
Method Blank	One per batch*
Instrument Blank	As needed
Blank Spike	One per batch*
Matrix Spike	One per batch*
Matrix Spike Duplicate	One per batch*
Surrogate	Every sample and standard

*The maximum number of samples per extraction batch is twenty or per project specification.

11.2 Initial Calibration.

11.2.1 Refer to Section 11.1.

11.3 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard.

11.3.1 Refer to Section 11.2.

11.4 Continuing Calibration Verifications.

11.4.1 Refer to Section 11.3.

11.5 Method Blank.

11.5.1 The method blank is either DI water or acidified sodium sulfate (depending upon the sample matrix) which must be extracted and analyzed with each set of 20 or less samples. For a running batch, a new method blank is required for each different extraction day. The blanks are processed through all steps required for sample preparation and measurement.

11.5.2 Corrective action for non-compliant blanks must be taken if contamination is detected that interferes with detection of targeted analytes. If the method blank contains a target analyte above its MDL, the entire batch must be re-extracted and re-analyzed.

11.5.3 Surrogate compounds are added to all method blanks prior to extraction and analysis. If the surrogate accuracy in the blank does not meet criteria, the batch must be re-extracted and re-analyzed.

11.6 Blank Spike.

11.6.1 A blank spike must be extracted and analyzed with each set of 20 or less samples. For a running batch, a new blank spike is required for each different extraction day. The blank spike consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. It is spiked with the target analytes at a detectable concentration, usually the mid-point concentration of the calibration range between 20 ug/l and 10 000 ug/l for all compounds (see STD 4 in Table 3B).

11.6.2 The blank spike recoveries and surrogates should be assessed using current in-house limits specified in and programmed into the LIMS. If a blank spike is out of control, the following corrective actions must be taken and all the associated samples must be re-extracted and reanalyzed. The exception is if the blank spike recovery is high and no hits reported in associated samples and QC batch. In that case, the sample results can be reported with footnote (remark) and no further action is required.

11.6.2.1 Check to be sure that there are no errors in the calculations, or spike solutions. If errors are found, recalculate the data accordingly.

11.6.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample batch.

11.6.2.3 If no problem is found, re-extract and reanalyze the sample batch.

11.6.3 When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the BS results are used to verify that the laboratory can perform the analysis in a clean matrix.

11.7 Matrix Spike/Matrix Spike Duplicate.

11.7.1 For every batch of 20 or less samples, a pair of matrix spike and matrix spike duplicate must be prepared and analyzed. Matrix spike and matrix spike duplicate are replicate sample aliquots to which a surrogate compound and a known amount of matrix spike compounds are added resulting in final concentration range between 20 ug/l and 10 000 ug/l for all compounds (see STD 4 in Table 3B). The MS and MSD are then extracted and run through any clean-up procedures along with the other samples in that batch to monitor the precision and accuracy of the extraction.

11.7.2 Assess the matrix spike recoveries and relative percent deviation (RPD) against the control limits.

11.7.3 If the matrix spike accuracy of any individual compound is out of control, the accuracy for the compound in the blank spike must be within control. Matrix interference is assumed and the data is reportable. No further corrective action is required.

11.8 Surrogate.

11.8.1 2,4-Dichlorophenylacetic acid (DCAA) is used as a surrogate standard to monitor the efficiency of the extraction and clean-up procedures. A known amount of surrogate standard is added to each sample including the QCs prior to extraction. The surrogate recovery data from individual samples must be evaluated versus the surrogate control limits developed by the laboratory.

11.8.2 If surrogate recovery is not within established control limits, corrective action must be performed if surrogate recoveries indicate that a procedural error may have occurred during the analysis of the sample.

11.8.2.1 Check the surrogate calculations for calculation or integration errors and perform corrections if detected.

11.8.2.2 Re-analyze the extract if calculation errors are not detected. If the surrogate recoveries for the re-analyzed extract are in control, report data from the re-analysis only.

11.8.2.3 If data from the reanalysis is also out of control, re-extract and reanalyze the sample.

11.8.2.4 If, upon reanalysis, the surrogate recoveries are acceptable, report the reanalysis data. If the recovery is again not within limits, the problem is considered to be matrix interference. Submit both data sets with the original analysis being reported.

11.8.3 The retention time shift for surrogate must be evaluated after the analysis of each sample. The sample must be reanalyzed when the retention time of any surrogate compound is outside the retention window.

11.8.3.1 Reanalysis may not be required for samples having visible matrix interference, defined as excessive signal levels from target or non-target interfering peaks. Check with the team leader or supervisor before making any decisions. .

11.9 Instrument Blank. A system evaluation sample containing hexane surrogate standards added. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples. Instrument Blanks may be analyzed after CCVs and as determined by the analyst.

11.10 Calculations

11.10.1 Calculation Factor (CF)

$$CF = \frac{A_s}{C_s}$$

where: A_s = Area of the peak for the compound being measured.
C_s = Concentration of the compound being measured (μg/l).

11.10.2 Percent Relative Standard Deviation (% RSD)

$$\%RSD = \frac{SD}{CF_{av}} \times 100$$

where: SD = Standard Deviation.
CF_{av} = Average calibration factor from initial calibration.

11.10.3 Percent Difference (% D)

$$\% D = \frac{|CF_{av} - CF_c|}{CF_{av}} X\ 100$$

where: CF_c = CF from continuing calibration (CBCHK).

11.10.4 Concentration (Conc.).

11.10.4.1 For water: without correction factor

$$Conc.\ (\mu g/l) = \frac{A_c \times M}{CF_{av}}$$

$$M = \frac{V_f \times D}{V_i}$$

11.10.4.2 For water: with correction factor

$$Conc.\ (\mu g/l) = \frac{A_c \times M \times MW_{acid}}{CF_{av} \times MW_{ester}}$$

$$M = \frac{V_f \times D}{V_i}$$

11.10.4.3 For soil/sediment (on a dry weight basis): without correction factor

$$Conc.\ (\mu g/kg) = \frac{A_c \times M}{CF_{av}}$$

$$M = \frac{V_f \times D}{W_s \times S}$$

11.10.4.4 For soil/sediment (on a dry weight basis): with correction factor

$$Conc.(\mu g/kg) = \frac{A_c \times M \times MW_{acid}}{CF_{av} \times MW_{ester}}$$

$$M = \frac{V_f \times D}{W_s \times S}$$

where:
A_c = Area of peak for compound being measured.
V_f = Final Volume of total extract (ml).
D = Secondary dilution factor.

V_i = Initial volume of water extracted (ml).
W_s = Weight of sample extracted (g).
S = (100 - % moisture in sample) / 100 or % solid/100.
M = Multiplier
MW_{acid} = Molecular weight of the herbicide acid
MW_{ester} = Molecular weight of the herbicide methyl ester

11.10.5 Percent Recovery (% R)

$$\% R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

11.10.6 Relative Percent Difference (RPD)

$$RPD = \frac{|C_1 - C_2|}{(1/2)(C_1 + C_2)} \times 100$$

where:
C_1 = Matrix Spike Concentration or the result on column 1.
C_2 = Matrix Spike Duplicate Concentration or the result on column 2.

12.0 Documentation

12.1 Record the analytical sequence in the instrument logbook prior to initiating the analysis.

12.1.1 Document reasons for reanalysis in the comments section of the log book.

12.2 Record all standards preparation data in the standards logbook. All columns must be completed and countersigned by a supervisor. Cross-reference the SGS lot number on the standard vial.

12.3 The instrument maintenance logbook must be completed daily. There is a separate logbook for each instrument.

12.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

12.5 Unused blocks of any form must be X'ed and Z'ed by the analyst before submitting the data for review.

12.6 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information should also be verified during this review.

13.0 Data Review & Reporting

13.1 Initial and continuing calibration check. Verify that all calibration and continuing calibration criteria have been achieved. If the criteria had not been achieved, corrective action must be performed to bring the system in control before analyzing any samples.

13.1.1 If samples had been analyzed under non-compliant calibration criteria, all sample extracts must be re-analyzed once the system is brought into control.

13.2 Quality Control Data Review. Review all QC data. If QC criteria were not achieved, perform corrective action before proceeding with analysis.

13.2.1 In some situation, corrective action may demand that the entire sample batch be re-extracted and re-analyzed before processing data.

13.3 Chromatogram Review. The chromatogram of each sample is evaluated for target compounds.

13.3.1 Check specific retention time windows for each target compound for the presence of the target compound in each chromatogram.

13.3.1.1 Each sample may require the reporting of different target compounds. Review the login to assure that the correct target compounds are identified.

13.3.2 The compound must be identified on the primary and confirmatory column before assigning a qualitative identification.

13.3.3 Manual integration of chromatographic peaks are given electronic signatures after supervisor review. The before and after scans are saved for the ICV, CCV and projects that require this specifically.

13.4 Transfer to LIMS. Following the initial screen review, transfer the processed data to the LIMS.

13.4.1 Print the processed data and compare the printed values to the original values to verify transfer accuracy.

13.4.2 If transfer errors occurred, the error s must be corrected before the data is re-submitted.

13.5 Hardcopy Print & Data Package Assembly.

13.5.1 After successful transfer is verified, the analyst approves the data.

13.5.2 The supervisor performs the final review and releases the data for reporting.

14.0 Pollution Prevention & Waste Management

14.1 Pollution Prevention. Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 18.2.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non hazardous aqueous wastes.
14.2.2 Hazardous aqueous wastes.
14.2.3 Chlorinated organic solvents.
14.2.4 Non-chlorinated organic solvents.
14.2.5 Hazardous solid wastes.
14.2.6 Non-hazardous solid wastes.

15.0 References

15.1 Test Methods for Evaluating Solid Wastes, Physical Chemical Methods (SW846) Third Edition All Updates, January 1998.

15.2 Code of Federal Regulations (CFR) 40, Parts 136 - Revised as of July 1, 1995, Appendix B-Definition and Procedure for the Determination of the Method Detection Limit, Revision 1.11.

Table 1. Gas Chromatograph Conditions

Instrument*:	HP-5890, HP-6890
Detector:	Electron Capture Detectors
Column:	A – CLP I, 30m x 0.32mm
	B – CLP 2, 30m x 0.32mm
Carrier:	Helium
Makeup:	5% Methane in argon
Temp. Program*:	65°C for 2 min.,
	then 20°C /min. to 175°C
	then A – 2.5°C /min. to 200°C
	B – 20°C /min. to 280°C (hold 4 mins)
Injection Volume:	1 μL, splitless
Injector Temp.:	225°C
Detector Temp.:	300°C

* Parameter modification allowed for performance optimization provided operational and QC criteria is achieved.

Table 2. Intermediate Calibration Standard Solution					
Compound Name	Stock mg/l	Volume Stock ul	Final Vol. in Hexane ml	Final Conc. (ester) ug/l	Final Conc. (acid) ug/l
2,4-D	200	100	10	2000	1880
2,4-DB	200	100	10	2000	1894
2,4,5-TP (Silvex)	200	20	10	400	380
2,4,5-T	200	20	10	400	379
Dalapon	200	20	10	400	364
Dicamba	200	20	10	400	376
Dichloroprop	200	100	10	2000	1888
Dinoseb	200	100	10	2000	1890
MCPA	2000	500	10	100 (mg/l)	935(mg/l)
MCPP	2000	500	10	100 (mg/l)	939(mg/l)
Pentachlorophenol	200	10	10	200	190
Or Pentachlorophenol	100	20	10	200	190
Picloram	200	100	10	2000	1890
Or Picloram	100	200	10	2000	1890
2,4 Dichlorphenylacetic acid (s)	100	400	10	4000	3744

(s) = Surrogate

Table 3A. Initial Calibration Standards Preparation Scheme		
Standard Solution	Volume Intermediate (ul)	Final Volume in Hexane (ml)
STD 1	250	1
STD 2	200	1
STD 3	150	1
STD 4	100	1
STD 5	50	1
STD 6	25	1

Table 3B. Initial Calibration Standard Concentrations						
Compound Name	Concentration ug/l (acid)					
	Std 1	Std 2	Std 3	Std 4	Std 5	Std 6
2,4-D	470	375	282	188	94	47
2,4-DB	474	379	284	189	95	47
2,4,5-TP (Silvex)	95	76	57	38	19	9.5
2,4,5-T	95	76	57	38	19	9.5
Dalapon	91	73	55	36	18	9.1
Dicamba	94	75	56	38	19	9.4
Dichloroprop	472	378	283	189	94	47
Dinoseb	473	378	284	189	95	47
MCPA	23375	18700	14025	9350	4675	2338
MCPP	23475	18780	14085	9390	4695	2348
Pentachlorophenol	48	38	29	19	9.5	4.8
Picloram	473	378	284	189	95	47
2,4 Dichlorphenylacetic acid (s)	936	749	562	374	187	94

Table 5 ICV -Second Source Calibration Check Standard					
Compound Name	Intermediate Conc. µg/l	Volume Added µl	Final Vol. in Hexane ml	Final Conc. (ester)µg/l	Final Conc. (acid)µg/l
2,4-D	2000	150	1	300	282
2,4-DB	2000			300	284
2,4,5-TP (Silvex)	400			60	57
2,4,5-T	400			60	57
Dalapon	400			60	55
Dicamba	400			60	56
Dichloroprop	2000			300	283
Dinoseb	2000			300	284
MCPA	100 (mg/l)			15000	14025
MCPP	100 (mg/l)			15000	14085
Pentachlorophenol	200			30	29
Picloram	2000			300	284
2,4 Dichlorphenylacetic acid (s)	4000			600	56

(s) = Surrogate

Table 6 Reporting Limits (*)(acid)		
Compound	Water	Soil* based on 35g
	µg/l	µg/kg
2,4-D	0.47	14
2,4-DB	0.47	14
2,4,5-TP (Silvex)	0.095	2.8
2,4,5-T	0.095	2.7
Dalapon	0.091	2.6
Dicamba	0.094	2.7
Dichlorprop	0.47	14
Dinoseb	0.47	14
MCPA	47	1403
MCPP	47	1409
Pentachlorophenol	0.048	1.3

(*) Soil RLs will be increased when percent solids values are incorporated into the calculation.
Water RLs will be increased when sample volume is limited.

Table 7			
Ester to Acid Correction Factors			
Herbicide	MW Acid	MW ester	Correction factor
Dalapon	143	157	0.911
Dicamba	221	235	0.940
Dichloroprop	235	249	0.944
2,4-D	221	235	0.940
2,4-DB	249	263	0.947
2,4,5-T	255.5	269.5	0.948

2,4,5-TP	269.5	283.5	0.951
Dinoseb	240	254	0.945
Picloram	241.5	255.5	0.945
Pentachlorophenol	266.5	280.5	0.950
MCPA	200.5	214.5	0.935
MCPP	214.5	228.5	0.939

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
10.2.6	Added TNI acceptable procedure to delete points from an ICAL
3.2	Updated MDL reference to 40CFR 136,APPx B, Rev 2
4.0	Updated MDL definition

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed
3.2.1-3.2.2	Replaced by new MDL procedure.See 3.2

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
20	03/25/2020	Maria Ruschke

END OF DOCUMENT

LAB SUPERVISOR: _____

QA OFFICER: _____

EFFECTIVE DATE: 12/15/21

TITLE: HEXAVALENT CHROMIUM (SOILS)
REFERENCES: SW846 3060A (Revision 1, Dec. 1996) - for digestion;
SW846 7196A (Revision 1, July 1992) - for analysis
REVISED SECTIONS: 11.7.1 (%RE TNI 2016); 3.2; 5.1; Format

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of hexavalent chromium in soils, sludges, brick, concrete, and other solid matrices. The solid sample is digested in an alkaline digestion solution to solubilize both water soluble and water insoluble hexavalent chromium compounds. Magnesium chloride in a phosphate buffer is added to suppress oxidation of Cr (III). The hexavalent chromium is determined in the digestate by reaction with diphenylcarbazide in acid solution. The diphenylcarbazide complex produces a characteristic pink color which can be measured spectrophotometrically at 540 nm.

2.0 SUMMARY OF METHOD

2.1 This method uses an alkaline digestion to solubilize both water-insoluble (with the exception of partial solubility of barium chromate in some soil matrices, and water soluble Cr(VI) compounds in solid waste samples. The pH of the digestate must be carefully adjusted during the digestion procedure. Failure to meet the pH specifications will necessitate redigestion of the samples.

2.2 The sample is digested using 0.28M Na2CO3 /0.5M NaOH solution and heating at 90- 95°C for 60 minutes to dissolve the Cr(VI) and stabilize it against reduction to Cr(III).

2.3 The Cr(VI) reaction with diphenylcarbazide is the most common and reliable method for analysis of Cr(VI) solubilized in the alkaline digestate. The use of diphenylcarbazide has been well established in the colorimetric procedure, in rapid-test field kits, and in the ion chromatographic method for Cr(VI). It is highly selective for Cr(VI) and few interferences are encountered when it is used on alkaline digestates.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at 0.40 mg/kg for soils, based on the low standard of 0.010 mg/l x 100 ml volume with a sample weight of 2.5 g.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B, Rev. 2. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Process all raw data for the replicate analysis in each MDL study. Forward the processed data to the QA group for archiving.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD. The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard be run at a frequency of approximately 10 percent. (For some methods this is mandatory and for some it is a recommendation only. Refer to individual method SOP's) For most methods, the mid-level calibration check standard criteria is + 10 percent of the true value. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the detection limit.

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. Assess laboratory performance against the control limits specified in the SOP. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent should be used.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to generate limits usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent should be used.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result}) \times 100}{(\text{Amount Spiked})} = \text{Matrix Spike Recovery}$$

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MPLS). The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLs should be determined approximately once per year for frequently analyzed parameters.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS-Dayton Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard.

5.3 Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.4 The following analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: hexavalent chromium.

5.5 For weighing out hexavalent chromium samples and standards, put clean paper towels around weighing area; front and both sides of analytical balance. These will be used to capture possible spills or dust. Place an additional clean paper towel to the side for cleaned items after weighing such as spatula, weighing dish and similar.

5.6 Before weighing, put on a dust mask. After taking a dust mask, reseal the plastic bag of dust masks for the next person. Hands must be clean.

5.7 Keep all standards in a secondary container while in use to contain in case of spills. Place the secondary container so that it will not interfere with using the balance.

5.8 Put on nitrile gloves and carefully weigh out samples and standards, putting lids on immediately after use. For the solid LCS (NIST), make sure to mix the standard in the fume hood by stirring. This LCS should not be shaken to mix. Standard must be in a closed container when outside the fume hood except for when you are weighing out your aliquot. Take out the aliquot and immediately reclose the container.

5.9 Rinse all spatulas and weighing dishes with 10% nitric after use. A final rinse with DI water will follow. Place cleaned items on the clean paper towel.

5.10 All rinse must be captured in a properly labeled poly container. Paper towels and gloves to be disposed in lab waste receptacles.

5.11 Put a small amount of 10% nitric on the capture paper towels. Fold paper towels at least once and use to wipe down the area, including the balance to leave it clean for the next person. Then wet paper towels with Dl water and wipe off the acid residue.

5.12 Remove nitrile gloves inside out and replace with a clean pair. Return cleaned items and utensils to their appropriate storage bag for the next person.

5.13 Store all items in a labeled cabinet.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIME

6.1 Soil samples should be kept under refrigeration at ≤ 6°C until time of digestion.

6.2 All samples that are analyzed following this method must be analyzed within 30 days of sample collection. The alkaline digestate is stable for up to 168 hours after extraction from soil

7.0 INTERFERENCES

7.1 Waste material suspected of containing soluble Cr(lll) concentrations greater than 4 times the laboratory Cr(VI) reporting limit may have Cr(VI) results that are biased high due to method induced oxidation. The addition of Mg (II) salts, in a phosphate buffer, is added to suppress this oxidation.

7.2 During the analysis of the alkaline digest for hexavalent chromium using diphenylcarbazide, high concentrations of molybdenum and mercury (> 200 mg/l) can interfere. Concentrations of vanadium greater than 10 times the level of hexavalent chromium may also cause interferences.

8.0 APPARATUS

8.1 Volumetric flasks and pipets and graduated cylinders, class A. All glassware should be washed with soap and tap water and then well rinsed with deionized water.

8.2 50 and 250 ml glass beakers with watch glasses. All glassware should be washed with soap and tap water and then well rinsed with deionized water.

8.3 Filter paper, 0.45 urn. Acceptable filter papers include the following: MSI cellulostic white grid filters, 0.45 urn, 47 mm (catalog number E04WG047S1).

8.4 Filter pump and vacuum filtration apparatus.

8.5 pH meter.

8.6 Hot plate, capable of maintaining the digestion solutions at 90 to 95 C, with constant stirring ability.

8.7 Four place analytical balance.

8.8 Thermometer, calibrated to an NIST certified thermometer a minimum of once per year

8.9 Graduated plastic beakers.

8.10 One or two place balance.

8.11 Spectrophotometer measuring at 540 nm and having a light path of 1-cm or longer. Model: Thermo Scientific GENESYS 20 UV-VIS Spectrophotometer, or similar.

9.0 REAGENTS

9.1 All reagents should be made from ACS grade reagents unless otherwise noted. Deionized water should be used whenever water is needed. The expiration date for standards and reagents is the date supplied by the manufacturer or if no expiration date is given, a default of 6 months is used. For acid solutions (nitric, sulfuric, hydrochloric) the expiration date is 2 years from the date of preparation of the solution.

9.2 Nitric acid, HNO3, concentrated, trace metals grade

9.3 Nitric acid, HNO3 5.0 M, trace metals grade. Add 32 ml of concentrated nitric acid to approximately 50 ml of Dl water. Dilute to a final volume of 100 ml with Dl water and mix well. Store at 20-25°C in the dark. Do not use concentrated nitric acid to make up the 5.0 M solution if it has a yellow tinge. The yellow color is indicative of a photo reduction of nitrate to nitrite, a reducing agent for Cr(VI).

9.4 Sodium Carbonate, Na2C03, anhydrous.

9.5 Sodium Hydroxide, NaOH.

9.6 Magnesium Chloride, MgCI2 (anhydrous). Note: 392.18 mg of MgCI2 is equivalent to 100 mg of Mg^{2+}.

9.7 Phosphate Buffer Solution (0.5 M K_2HPO_4/0.5 M KH_2PO_4 buffer at pH 7): Dissolve 87.09 g of KH_2PO_4 and 68.04 g of KH_2PO_4 into 700 ml of distilled deionized water. Transfer to a 1 liter volumetric flask and dilute to volume.

9.8 Digestion Solution: Dissolve 20.0 g of NaOH and 30.0 g of Na2CO3 in distilled deionized water in a one-liter volumetric flask and dilute to the mark. Store the solution in a tightly capped polyethylene bottle at 20 to 25°C and prepare fresh monthly. The pH of this digestion solution must be checked and recorded before using. If the pH is not greater than or equal to 11.5, then the digestion solution should be discarded and a new solution should be made up.

9.9 Insoluble hexavalent chromium spike, lead chromate, PbCrO4. The insoluble matrix spike is prepared by adding 10 to 20 mg of PbCrO4 to the insoluble matrix spike aliquot.

9.10 Soluble hexavalent chromium spiking solution stock. A 1000 mg/l stock solution of potassium dichromate can be used as the stock solution for the spiking solution. (Available as 1000 mg/l chromium solution, AAS grade from Fisher or equivalent).

9.11 Soluble hexavalent chromium spiking solution, 100 mg/l. Add 10.0 ml of the 1000 mg/l hexavalent chromium to a 100 ml volumetric flask and dilute to volume with Dl water. Mix well. One (1.00) ml of this spiking solution can be used to spike the soluble matrix spike aliquot. The approximate level of the spike in the spiked sample will be 40 mg/kg.

9.12 Sulfuric acid, 10 percent (v/v). Add 10 ml of concentrated sulfuric acid to approximately 70 ml of Dl water. Mix well and let cool. Dilute to a final volume of 100 ml with Dl water.

9.13 Acetone. Do not use acetone that comes in a container with a metal or metal lined cap.

9.14 Diphenylcarbazide solution. Dissolve 0.250 g of 1,5 diphenylcarbazide in 50 ml of acetone. Store in a brown or a foil covered bottle to minimize exposure to light. Discard when the solution becomes discolored or monthly, whichever comes first. (Note: Be sure to check the quality of the diphenylcarbazide solution before adding it to the sample.

9.15 Hexavalent Chromium Calibration Standard Solutions. The calibration standards must be prepared fresh daily before digestion, but may be associated with multiple digestion batches from the same day. For instrument calibration, prepare the standards from the stocks as shown below. For all standards, add 50 ml of digestion solution to a labeled plastic beaker and then pipet in the appropriate amount of a stock solution. Do not dilute these standards to the final volume at this time. All calibration standards must go through the entire digestion process, starting at step 10.4.

9.15.1 Hexavalent Chromium, 10 mg/l stock solution. Add 1.00 ml of 1000 mg/l hexavalent chromium to a 100 ml volumetric flask and dilute to volume with Dl water. Mix well.

9.15.2 Hexavalent Chromium, 1.0 mg/l stock solution. Add 10 ml of 10 mg/l hexavalent chromium to a 100 ml volumetric flask and dilute to volume with Dl water. Mix well.

9.15.3 Add the amount of stock specified below to the 50 ml of digestion solution.

Blank: No spike is added to the blank.
0.010 mg/l: Add 1.- mg of 1.100 mg/l
0.050 mg/l: Add 0.50mg of 10.0 mg/l
0.100 mg/l: Add 1.00 ml of 10.0 mg/l
0.300 mg/l: Add 3.00 ml of 10.0 mg/l
0.500 mg/l: Add 5.00 ml of 10.0 mg/l
0.800 mg/l: Add 8.00 ml of 10.0 mg/l
1.00 mg/l: Add 10.0 ml of 10.0 mg/l

9.16 Hexavalent Chromium CCV (Continuing Calibration Verification) Solutions. The check standards must be prepared fresh daily before digestion. Prepare the standards from the stocks as shown below. All check standards must go through the entire digestion process, starting at step 10.4. A minimum of 4 check standards should be made for a batch of 20 samples. Note: The check standards must be made from a different source than the calibration standards.

9.16.1 Hexavalent Chromium 10.0 mg/l stock solution. Add 2.00 ml of 1000 mg/l hexavalent chromium to a 200 ml volumetric flask and dilute to volume with Dl water. Mix well.

9.16.2 For initial calibration curves that have the 1.00 mg/l standard as the upper limit, a calibration check at 0.500 mg/l must be used. Therefore, add the amount of stock solution specified below to 50 ml of digestion solution. Do not dilute to a final volume. This entire solution should be digested.

0.500 mg/l: Add 5.00 ml of the 10.0 mg/l stock solution

10.0 DIGESTION PROCEDURE

Below is a step-by-step procedure for the digestion of samples for the determination of hexavalent chromium.

10.1 For each sample to be analyzed, weight out 2.5 + 0.10 g of the sample into a clean, labeled glass beaker. A one or two place balance may be used for this weighing. The sample should be well mixed before the aliquot is removed as described in OQA-042, the representative sample aliquot SOP.

10.1.1 For the sample that is to be used for the quality control sample, weigh out six 2.5 + 0.10 g aliquots from the well mixed sample. One aliquot will be for the soluble Cr(VI) matrix spike, one aliquot will be for the insoluble Cr(VI) matrix spike, one aliquot will be for the original sample analysis, one aliquot will be for the duplicate sample analysis, and the remaining two aliquots will be used to complete the procedures required if the initial post-digest spike does not meet the + 15 criteria.

10.2 Add the spikes to the matrix spikes and the spike blanks.

10.2.1 Spike the soluble Cr(VI) matrix spike with 1.0 ml of the 100 mg/l Cr(VI) spiking solution. (Check with the area supervisor or manger before starting to see if an additional spike level will be needed.)

10.2.2 Spike the soluble Cr(VI) spike blank with 1.0 ml of the 100 mg/l Cr(VI) spiking solution.

10.2.3 Using an analytical balance, weigh out 0.010 to 0.020 g of $PbCrO_4$ onto a clean piece of weighing paper and carefully add the spike into the insoluble matrix spike sample. Make sure to record the weight used.

10.2.4 Note: This second spike blank is optional. Using an analytical balance, weigh out 0.010 to 0.020 g of $PbCrO_4$ onto a clean piece of weighing paper and carefully add the spike into the insoluble spike blank sample. Make sure to record the weight used.

10.3 Add 50 ml of digestion solution to each sample. Also add approximately 0.4 g of $MgCl_2$(from 0.38 to 0.42 g) using two digits analytical balance. After that, using a repipetter, add 0.5 ml of the 1.0 M phosphate buffer. In addition to the samples, 3 extra beakers should be prepared for the method blank, the soluble Cr(VI) spike blank, and the insoluble Cr(VI) spike blank.

10.4 In addition to the samples, the CCV (continuing calibration check) standards and calibration standards should also be digested. Add the entire CCV solution (refer to step 9.16) into a

clean, labeled glass beaker. Also add approximately 0.4 g of MgCI2 and 0.5 ml of the 1.0 M phosphate buffer.

10.5 Cover all samples and quality control (including the calibration check samples) with watch glasses. Add a stirring bar to each sample and stir the samples for at least 5 minutes without heating.

10.6 Place the samples on a stirring hot plate. Heat the samples up to 90 - 95°C with constant stirring for 60 minutes, maintaining a temperature range of 90 to 95°C. The temperature should be measured by placing a calibrated thermometer in an extra beaker containing digestion reagent on the hot plate. The temperature must be recorded at 30 minutes and 60 minutes during the digestion process. Both the start and the stop time of the digestion must be recorded.

10.7 Allow digestates to cool to room temperature, while continuing to stir or agitate the samples. Filter them through 0,45 urn filter paper. Rinse the filter and filtration apparatus with Di water and transfer the filtrate into labeled graduated plastic beakers.

10.7.1 If the filters become clogged using the 0.45 urn filter paper, a larger size filter paper (Whatman GFB or GFF) may be used to pre-filter the samples. However, the final filtration must be through the 0.45 urn filters. If a pre-filtration is required, it should be recorded on the digestion log.

10.7.2 The solids and the filter remaining after the filtration of the matrix spikes may need to be saved in a labeled plastic beaker and stored in the refrigerator. If low recoveries are obtained on the matrix spikes, these solids may be needed for additional analyses Check with the area supervisor or manager for further instructions.

10.7.3 At this point, the digestates are stable and may be held for up to 168 hours before proceeding with step 10.8.

10.8 Do not start this step unless the analysis will be started within one hour after this step has been completed. The calibration standards should also be taken through this process,

10.8.1 Place a stirring bar in the sample and place it on a stirring plate. Adjust the pH of the solution between 7.00 and 8.00 by carefully adding 5.0 M nitric acid to the digestate while constantly measuring the pH. Do not let the pH of the solution go below 7.00. If the pH goes below 7.00, then the digestate must be discarded and a new digestate prepared. Make sure to record the final pH.

10.8.2 If the pH is changing too rapidly with 5.0 M nitric acid, then a more dilute solution of nitric acid may be used for the pH adjustment.

10.8.3 Carbon dioxide and nitric acid fumes will be evolved during this process. Therefore, this step must be performed in a hood or well-ventilated area.

10.9 Quantitatively transfer the contents of the beaker to a 100 ml volumetric flask or class A graduated cylinder and adjust the sample volume to the mark with DI water. Mix well. At this point, a brief description of each sample (color, turbidity, etc.) can be added to the digestion log

10.9.1 If the same cylinder is used for multiple samples, it must be rinsed with deionized water at least 3 times between samples.

11.0 ANALYSIS PROCEDURE

11.1 Turn on the spectrophotometer and let it warm up for at least 30 minutes. Set the wavelength to 540 nm and adjust the zero.

11.2 Using a class A graduated cylinder, transfer quantitatively 45.0 ml of the sample or standard to be analyzed to a labeled plastic beaker.

11.3 Add 1.0 ml of diphenylcarbazide solution and mix well.

11.4 Slowly add 10 percent sulfuric acid to each sample, mixing well after each addition. Adjust the pH to a range of 1.5 to 2.5. Test the pH of each sample with a pH meter when the effervescence is minimal and record this reading. (On some samples, a small amount of effervescence has been observed several hours after the pH adjustment was completed.)

11.4.1 A background correction point must also be prepared for each sample with 10 ml of sample adjusted to a pH of 1.5 to 2.5 with sulfuric acid. The background correction point should not contain diphenylcarbazide. Make sure to record the adjusted pH of the background correction point.

11.5 If the samples are turbid at this point, filter them through a 0.45 urn filter. If the sample aliquot is filtered, the background aliquot must also be filtered.

11.6 Transfer the samples to 50 ml volumetric flasks or class A graduated cylinders and dilute to a final volume of 50 ml with Dl water. Let the samples stand for 5 to 10 minutes after the reagents are added for full color development.

11.6.1 If the same cylinder is used for multiple samples, it must be rinsed with deionized water at least 3 times between samples.

11.7 Read the standard calibration curve first, and then a calibration check standard and a reagent blank, making sure to record all results on the strip chart recorder. The correlation coefficient for the curve must be greater than or equal to 0.995, the check standard must be within 10 percent of the true value, and the reagent blank must be less than the reporting detection limit before the analysis can be continued.

$$r = \frac{\Sigma(x - \bar{x})(y - \bar{y})}{\sqrt{\Sigma(x - \bar{x})^2 \Sigma(y - \bar{y})^2}}$$

Where r = correlation coefficient
x = amount of analyte
y = response of instrument

$\bar{x}$ = average of x values

$\bar{y}$ = average of y values

11.7.1 Additionally, calculate and document a measure of relative error in the calibration. For this method the result must be +/- 30%. If the criterion is not met the curve must be reanalyzed.

11.7.1.1 For calibrations evaluated using an average response factor, the determination of the relative standard deviation (RSD) is the measure of the relative error.

11.7.1.2 For calibrations evaluated using correlation coefficient or coefficient of determination, the laboratory shall evaluate relative error by either Relative Error (%RE) or Relative Standard Error (%RSE).

11.7.1.2.1 Relative Error (%RE) is calculated using the following equation:

$$\%\ Relative\ Error = \frac{x'_i - x_i}{x_i} \times 100$$

xi = True value for the calibration standard
x'i = Measured concentration of the calibration standard

This calculation shall be performed for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level.

The Relative Error at both of these levels shall meet the criteria specified in the method

11.7.1.2.2 Relative Standard Error (%RSE) is calculated using the following equation:

$$\%\ RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x'_i - x_i}{x_i}\right]^2 \Big/ (n - p)}$$

xi = True value of the calibration level i
x'i = Measured concentration of calibration level i
p = Number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3)
n = Number of calibration points

11.8 After the curve and the initial quality control are completed, the samples may be analyzed. First read the sample result. If the result is over the highest point in the calibration curve, do not read the background correction point. If the result is within the calibration curve, the background correction point must be read immediately after the sample analysis is complete and before starting the next sample.

11.9 After every 10 samples or every 20 readings (10 samples plus 10 background correction points), a digested CCV and a reagent blank will be analyzed. The reagent blank must be less

than the reporting limit, and the CCV must be within 10% of the true value. If they are outside of this range, do not proceed. Check with the laboratory supervisor or manager for further directions.

11.10 After the quality control sample analysis is completed, prepare a post-digest spike on this sample. The sample should be spiked at 2 times the concentration found in the original sample aliquot or 40 mg Cr(VI)/kg, whichever is greater. Then proceed through steps 11.3 to 11.6 and analyze the spiked sample. Calculate the recovery immediately. If the recovery is not within 85 to 115 percent, proceed to steps 11.11 and 11.12.

11.10.1 The 40 mg/kg spike can be made by spiking a 45 ml aliquot of digestate containing 1.125 g of digested sample with 0.45 ml of 100 mg/l Cr(VI) standard (Section 9.10).

11.10.2 This spiking level requirement is taken from method 3060A. A lower level spiking requirement is given in the NJDEP 7196A method, but guidance from the state suggested using the 3060A spiking levels when following the 3060A digestion.

11.11 Dilute by a factor of 1:5 a fraction of the quality control sample. Place 45 ml of the diluted sample into a plastic beaker. Then proceed through steps 11.3 to 11.6 and analyze the sample. Also prepare a background correction point at this dilution and analyze it immediately following the analysis of the diluted sample.

11.12 Take an additional 45 ml aliquot of the sample and adjust the pH to between 8.0 and 8.5 using 1.0 N NaOH. Record the final pH. Then spike the sample at 2 times the concentration found in the original sample aliquot or 40 mg Cr(VI)/kg, whichever is greater. After the sample is spiked, proceed with steps 11.3 through 11.6, and analyze the pH adjusted post-digest spike.

11.13 Before completion of the analysis, a second analyst must verify that all pH adjustments are present and documented in the paperwork.

11.14 The calculations should be done as shown below. Values less than the IDL should be treated as zero for all calculations.

11.14.1 Calculation of sample result:

Conc. Cr(VI)in the sample in mg/kg =

$$\frac{\text{(conc. in digestate in ug/ml) x (final volume in ml) x DF}}{\text{(initial sample weight in g) x (\%solids/100)}}$$

11.14.1.1 For samples requiring trivalent chromium results, the following equation must be used:

Conc. Total chromium (mg/kg) - Conc. Hexavalent chromium (mg/kg)

11.14.1.2 Calculation of amount spike:

Spike amount (SA) in mg/kg =

$$\frac{\text{(conc. of spiking solution, µg/ml) x (vol. of spike, ml)}}{\text{(initial sample weight in g) x (\%solids/100)}}$$

11.14.1.3 Calculation of matrix spike recovery.

$$\text{MS Rec.} = \frac{\text{(SSR – SR) x 100}}{\text{SA}}$$

where SSR = Spiked sample result
SR = Sample result
SA = Spike added

11.14.1.4 Calculation of duplicate rpd.

$$\text{Dup RPD.} = \frac{\text{(SR - DR) x 100}}{\text{\{(SR + DR)/2\}}}$$

where SR = Sample result
DR = Duplicate result

12.0 QUALITY CONTROL

This section outlines the minimum QA/QC operations necessary to satisfy the analytical requirements as taken from these methods. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

12.1 A new 5 point calibration curve must be analyzed on each analysis day. The calibration curve must have a correlation coefficient greater than or equal to 0.995 percent. The calibration curve must be digested in the same manner as the samples.

12.2 All samples should initially be analyzed undiluted. If the sample concentration is higher than the highest standard, then the sample should be diluted and reanalyzed. The dilution should be made so that, if possible, the sample is in the mid-range of the calibration curve.

12.3 One preparation blank is required for each set of 20 samples or less or with each batch, whichever is most frequent. The preparation blank must contain all of the reagents in the sample volumes as used in the preparation of the samples. (The preparation blank should never be used to blank correct the samples.) The preparation blank must be less than the reporting limit. If the preparation blank does not meet the criteria, then the entire batch must be redigested.

12.4 A continuing calibration verification (CCV) standard at approximately the mid-point of the curve must be analyzed after every 10 samples or every 20 readings (10 sample readings plus 10 background readings). The CCV standard must be prepared from a different stock than the calibration curve and should be taken through the digestion process as outlined in the procedure section of this SOP. A CCV standard must be analyzed at the beginning of the

analysis immediately after the analysis of the calibration curve. All CCV standards must be within 10 percent of the true value for that standard. If they are outside of this range, do not proceed. Check with the laboratory supervisor or manager for further directions.

12.5 A reagent blank (or CCB) must be analyzed after each CCV. The reagent blank must be less than the reporting detection limit. If this criteria is not met do not proceed. Check with the laboratory supervisor or manager for further directions.

12.6 A duplicate sample must be prepared and analyzed for each set of 20 samples of a similar matrix or with each batch, whichever is smaller. An acceptance criteria of 20 percent relative percent difference should be applied if the original and duplicate sample values are greater than or equal to 4 times the reporting detection limit. If the values are less than 4 times the reporting detection limit, then a control limit of + the reporting detection limit should be applied.

12.7 Both a soluble and an insoluble hexavalent chromium matrix spike must be prepared and analyzed for each set of 20 samples of a similar matrix or with each batch, whichever is smaller. The acceptance range for matrix spike recoveries is 75 to 125 percent recovery. If the matrix spike recoveries for either the soluble or the insoluble spikes are not within these recovery limits, then the lab supervisor or manager must be immediately notified. The client services department will then be notified to contact the client. The method requires additional testing as listed below, but the lab should not proceed with this testing until client approval is obtained and the testing is logged into the LIMS system.

12.7.1 All samples and quality control must be re-homogenized, re-digested and reanalyzed to verify the original sample results.

12.7.2 Additional tests, such as oxidation-reduction potential, pH, sulfide, ferrous iron, etc., may be requested to help quantify the reducing nature of the sample. For some projects, eH and pH analysis may be specified for all samples at the start of the project. Eh and pH data plots must be provided in the data deliverable if this analysis is specified

12.7.3 A mass balance study for total chromium may be done, using the digested solids remaining after the alkaline digestion and filtration of the matrix spike and from a unspiked aliquot of the sample.

12.8 A post-digest spike must be prepared and analyzed for each set of 20 samples of a similar matrix or with each batch, whichever is smaller. The acceptance range for post digest spike recoveries is 85 to 115 percent recovery.

12.9 A spike blank or lab control sample must be prepared and analyzed for each set of 20 samples of a similar matrix or with each batch, whichever is smaller The spike blank or lab control can be prepared using either soluble or insoluble hexavalent chromium as the spike. The acceptance range for the spike blanks is 80 to 120 percent recovery. If the spike blanks are not within that range, then the entire batch must be redigested and reanalyzed.

12.10 Spike Witnessing. Addition of insoluble and soluble spike amounts must be verified by a spike witness at the time spike additions occur. These include all spiked blanks, matrix spikes, and if applicable, solid lab control standards. The spike witness must verify the correct solutions and amounts of those solutions are being added to the correct samples (or containers, if spiked

blanks). The spike witness must document (by initialing and dating) on the preparation log that they have observed and confirmed proper spike additions.

13.0 DOCUMENTATION REQUIREMENTS

13.1 The analyst should document all relevant information, including all sample weights and volumes, digestion times and temperatures, all intermediate and final pH values, all times relevant to the pH adjustment process, all sample and background analysis results, and any relevant comments for any section of the digestion or analysis. Sample digestion and analysis sheets are provided.

13.2 Analyses are done using an automated analysis spreadsheet where the sample absorbances are recorded electronically. If this electronic recording option is not available, then the analyst must verify all recorded absorbances. All reagent identification numbers should be recorded on the sample worksheets. In addition, all reagent information such as lot numbers should also be recorded in the reagent logbook.

14.0 DATA REVIEW AND REPORTING

14.1 All samples should be updated to analysis (GN) batches in the LIMS system. The analyst should calculate all matrix spike, duplicate, external, and CCV recoveries and review the results of all blanks.

14.2 All documentation must be completed, including reagent references and spike amounts and spiking solution references.

14.3 A data file should be exported to the LIMS system and the spike amounts should be entered into the file at the GNAPP process step.

14.4 A final data package, consisting of the prep and analysis raw data, the LIMS cover page, the reagent reference pages, and the QC summary pages must be turned into the area supervisor or other senior reviewer for review.

14.5 After review by the supervisor, the data is released in the LIMS for access to the clients. Review includes verifying the spike amounts, recoveries, and RPD's as well as the sample results and document such as all pH values.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes

16.0 ADDITIONAL REFERENCES

16.1 No additional references are required for this SOP.

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
All	Updated to most current format
5.1	Changed Accutest to SGS-Dayton

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed
3.2.1	No longer applicable

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added
11.7.1	% RE per 2016 TNI

History of Revisions

Version #	Date of Revision	Revised By
18	12/15/2021	Angela Lattanzio

END OF DOCUMENT

LAB SUPERVISOR: _____

QA OFFICER: Maria Luschke

EFFECTIVE DATE: 7-9-21

TITLE: HEXAVALENT CHROMIUM IN WATERS
REFERENCES: SW846 Method 7196A
REVISED SECTIONS: FORMAT

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of hexavalent chromium in aqueous matrices. The hexavalent chromium is determined in the sample by reaction with diphenylcarbazide in acid solution. The diphenylcarbazide complex produces a characteristic pink color that can be measured spectrophotometrically at 540 nm.

2.0 SUMMARY

2.1 Dissolved hexavalent chromium, in the absence of interfering amounts of substances such as molybdenum, vanadium, and mercury, may be determined colorimetrically by reaction with diphenylcarbazide in acid solution. A red-violet color of unknown composition is produced. The reaction is very sensitive, the absorbancy index per gram atom of chromium being about 40,000 at 540 nm. Addition of an excess of diphenylcarbazide yields the red-violet product, and its absorbance is measured photometrically at 540 nm.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at 0.010 mg/l.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B, Rev 2. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD: The calibration check standard is a mid-range calibration standard.

EXTERNAL CHECK STANDARD: The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards

EXTERNAL CHECK STANDARD: The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards

SPIKE BLANK OR LAB CONTROL SAMPLE: Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample must be assessed. The duplicate RPD is calculated as shown below.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

METHOD BLANK: The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required daily and for every 20 samples

METHOD DETECTION LIMITS (MDL): The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.)

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS-Dayton Chemical Hygiene Plan which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 The following analytes covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: hexavalent chromium.

6.0 PRESERVATION & HOLDING TIME

6.1 Refrigerate all samples at ≤6°C until the time of analysis.

6.2 The holding time for this method is 24 hours from the time of collection. Analyze all samples within 24 hours.

7.0 INTERFERENCES

7.1 The hexavalent chromium reaction with diphenylcarbazide is usually free from interferences. However, certain substances may interfere if the hexavalent chromium concentration is relatively low. Hexavalent molybdenum and mercury salts also react to form color with the reagent: however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 200 mg/L of molybdenum and mercury can generally be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of hexavalent chromium will not generally cause interference.

7.2 Iron in concentrations greater than 1 mg/L may produce a yellow color, but the ferric iron color is not strong, and difficulty is not normally encountered if the absorbance is measured photometrically at the appropriate wavelength.

7.3 Reducing substances such as organic matter or sulfides can convert hexavalent chromium to trivalent chromium. These reactions may occur in the natural environment and during the digestion and measurement processes.

8.0 APPARATUS

8.1 Spectrophotometer capable of measurement at 540 nm, providing a light path of 1 cm or longer.

8.2 50 ml graduated cylinder (class A) with a stopper.

8.3 Volumetric flasks, class A.

8.4 Volumetric pipets, class A, for standards preparation.

8.5 Filter paper, 0.45 um and 0.10 um. Acceptable filter papers include the following:

8.5.1 MSI cellulostic white grid filters, 0.45 um, 47 mm (catalog number E04WG047S1)

8.5.2 Porectics polycarbonate membrane filters, 0.10 um, 47 mm (catalog number 13010)

8.6 Filter pump and vacuum filtration apparatus.

8.7 pH meter. Orion pH/ISE Meter Model 710A or equivalent

8.8 Four-place analytical balance. Acculab LA-110 or equivalent.

8.9 Graduated Plastic Beakers.

8.10 One or two place balance. Ohaus Galaxy 4000 or equivalent.

9.0 REAGENTS

9.1 All chemicals listed below are reagent grade unless otherwise specified. Use deionized water whenever water is required. The expiration date for standards and reagents is the date supplied by the manufacturer or if no expiration date is given, a default of 6 months is used. For acid solutions (nitric, sulfuric, hydrochloric) the expiration date is 2 years from the date of preparation of the solution.

9.2 Sulfuric acid, 10 percent (v/v): Add 10 ml of concentrated sulfuric acid to approximately 70 ml of DI water. Mix well and let cool. Dilute to a final volume of 100 ml with DI water.

9.3 Diphenylcarbazide Solution: Dissolve 250 mg of 1,5 diphenylcarbazide in 50 ml of acetone. Mix well. Store in a brown bottle or in a foil wrapped container to protect the solution from the light. Discard when the solution becomes discolored or monthly, whichever comes first. (NOTE: Make sure to check the quality of the diphenylcarbazide solution before adding it to the samples.)

9.4 Acetone. Do not use acetone that comes in container with metal or metal-lined caps.

9.5 Hexavalent Chromium Stock Solution. A 1000 mg/L (as Cr(VI))stock solution of potassium dichromate can be used as the stock solution for the spiking solutions and calibration standards. (Available as 1000 mg/L chromium solution, AAS grade from Fisher.)

9.6 Hexavalent Chromium Calibration Standard Solutions: Prepare the calibration standards fresh daily or each time the analysis is run. For instrument calibration, prepare the standards from the stocks as shown below.

9.6.1 Hexavalent Chromium 5.00 mg/L stock solution. Add 1.00 ml of 1000 mg/L hexavalent chromium to a 200 ml volumetric flask and dilute to volume with DI water. Mix well.

9.6.2 Hexavalent Chromium 1.00 mg/L stock solution. Add 10.00 ml of 5.0 mg/L hexavalent chromium to a 50 ml volumetric flask and dilute to volume with DI water. Mix well.

9.6.3 Hexavalent Chromium 7.5 mg/L stock solution. Dilute 1.50 ml of the 1000 mg/L stock solution to a final volume of 200 ml with DI water and mix well. (For use for spiking.)

9.6.4 Add the amount of stock specified below to the 50 ml volumetrics and bring to a final volume of 50 ml with DI water.

Blank: No spike is added to the blank.
0.010 mg/l: Add 0.50 ml of 1.00 mg/L.
0.050 mg/L: Add 0.50 ml of 5.00 mg/L.
0.100 mg/L: Add 1.00 ml of 5.00 mg/L.
0.300 mg/L: Add 3.00 ml of 5.00 mg/L.
0.500 mg/L: Add 5.00 ml of 5.00 mg/L.
0.800 mg/L: Add 8.00 ml of 5.00 mg/L.
1.00 mg/L: Add 10.0 ml of 5.00 mg/L.
2.00 mg/L: Add 20.0 ml of 5.00 mg/L. (*)

(*) If the curve is more linear without the 2.00 mg/l standard, then do not use this standard.

9.7 Hexavalent Chromium Check Standard Solutions: Prepare the check standards fresh daily or each time the analysis is run. Prepare the standards from the stocks as shown below. NOTE: Prepare the check standards from a different source than the calibration standards.

9.7.1 Hexavalent Chromium 5.0 mg/L stock solution. Add 1.00 ml of 1000 mg/L hexavalent chromium to a 200 ml volumetric flask and dilute to volume with DI water. Mix well.

9.7.2 Add the amount of stock specified below to DI water and dilute to a final volume of 50 ml.

9.7.2.1 For initial calibration curves that have the 2.00 mg/L standard as the upper limit, a calibration check at 1.00 mg/L is used. Therefore, add the amount of stock solution specified below to DI water and dilute to a final volume of 50 ml:

1.00 mg/L: Add 10.00 ml of the 5.00 mg/L stock solution.

9.7.1.1 For initial calibration curves that have the 1.00 mg/L standard as the upper limit, a calibration check at 0.500 mg/L is used. Therefore, add the amount of stock solution specified below to DI water and dilute to a final volume of 50 ml.

0.500 mg/L: Add 5.00 ml of the 5.00 mg/L stock solution.

9.8 1.0 N Sodium Hydroxide, NaOH. Add 4.0 g of NaOH pellets to Di water and dilute to a final volume of 100 ml. Mix well.

10.0 ANALYSIS

10.1 Turn on the spectrophotometer and let it warm up for at least 30 minutes. Set the wavelength to 540 nm and adjust the zero.

10.2 Using a graduated cylinder, transfer quantitatively 45.0 ml of the sample or standard to be analyzed to a labeled plastic beaker.

10.3 Add 1.0 ml of diphenylcarbazide solution and mix well.

10.4 Slowly add 10 percent sulfuric acid to each sample, mixing well after each addition. Adjust the pH to a range of 1.5 to 2.5. Test the pH of each sample with a pH meter when the effervescence is minimal and record this reading. (No significant effervescence is expected for most aqueous samples, although this will be seen when analyzing soil digestates.)

10.5 If the samples are turbid at this point, filter them through a 0.45 um filter. If they are still turbid after this filtration, a 0.10 um filter may also be used.

10.6 Transfer the samples to 50 ml volumetric flasks or class A graduated cylinders and dilute to a final volume of 50 ml with DI water. Let the samples stand for 5 to 10 minutes after the reagents are added for full color development. If the sample volume is greater than 50 ml, make sure to correct for the volume difference in the calculations.

10.6.1 Make sure to look at the initial color development of the samples. If the sample initially forms a dark color which quickly starts to fade, this indicates that the sample contains a high level of hexavalent chromium and must be run on dilution.

10.6.2 If the same cylinder is used for multiple samples, it must be rinsed with deionized water at least 3 times between samples.

10.7 Read the standard curve first and then a calibration check standard and a reagent blank, making sure to record all results on the strip chart recorder. The curve correlation coefficient is required to be greater than or equal to 0.995 and the check standard is required to be within 10 percent of the true value before the analysis can be continued.

10.8 After the curve and initial quality control is completed, the samples may be analyzed. First read the sample result. If it is over the calibration curve, do not read a background correction point. If it is within the calibration curve, a background correction point is prepared with 10 ml of sample adjusted to a pH of 1.5 to 2.5 with sulfuric acid. Make sure to record the adjusted pH of the background correction point. Do not add diphenylcarbazide reagent to the background correction point. Note: If the sample was filtered, process the background correction sample through the same filtration procedure. Read the background correction point immediately after the sample analysis is completed.

10.9 After every 10 samples, every 20 readings (10 samples plus 10 background correction points) and at the end of the sample analysis sequence a digested calibration check and a reagent blank must be analyzed.

10.10 Prepare a Method blank per batch of 20 or less. The method blank consists of 50 ml of deionized water.

10.11 Prepare a spike blank per batch of 20 or less. Spike the blank with150ug Cr(VI)/L, (The 150 ug/L spike can be made by spiking 1.0 ml of 7.5 mg/L Cr(VI) standard to a final volume of 50 ml.) Then proceed through steps 10.3 through 10.6 and analyze the sample.

10.12 Prepare a matrix spike and a duplicate on at least one sample per batch of 20 or less. Spike the sample selected as the matrix spike sample at 2 times the concentration found in the original sample aliquot or 150ug Cr(VI)/L, whichever is greater. (The 150 ug/L spike can be made by spiking 1.0 ml of 7.5 mg/L Cr(VI) standard to a final volume of 50 ml.) Then proceed through steps 10.3 through 10.6 and analyze the sample. Calculate the recovery immediately. If the recovery is not within 85 to 115 percent, proceed to steps 10.13 and 10.14.

10.13 Dilute a fraction of the quality control sample 1:5 and place 45 ml of the sample into a plastic beaker. Then proceed through steps 10.3 through 10.6 and analyze the sample. Also prepare a background correction point at this dilution and analyze it.

10.14 Take an additional 45 ml aliquot of the sample and adjust the pH to between 8.0 to 8.5 using 1.0 N NaOH. Record the final pH. Then spike the sample at 2 times the concentration found in the original sample aliquot or 150 ug Cr(VI)/L, whichever is greater. After the sample is spiked, proceed with steps 10.3 through 10.6. If the sample volume is greater than 50 ml, make sure to correct for the volume difference in the calculations.

10.15 Analysis of new sample matrices, or where matrix interference cannot be suppressed using sample dilution requires analysis by the method of standard additions. Matrix interference is determined by inconsistent dilution results, and inconsistencies in analysis of total Cr. Analysts, and reviewers are required to report results with dilution RPD greater than 40%, and/or Cr(VI) results greater than total Cr results to department supervisors. The method of standard additions is evaluated by department supervisors.

10.15.1 See department supervisor for guidance on procedure of Method of standard additions located in method SW846 7000A section 8.7.

11.0 CALCULATIONS

11.1 Calculation of the sample result.

conc. of hexavalent chromium in the sample (mg/l) =

$$\frac{\text{(conc. in ext.(mg/l) x (final ext. vol.(ml)}}{\text{(initial sample vol. (ml))}}$$

11.1.1 For samples requiring trivalent chromium results, the following equation must be used:

Conc. Total chromium (mg/l) – Conc. Hexavalent chromium (mg/l)

11.2 Calculation of amount spiked.

Amount spiked of hexavalent chromium (SA)=

$$\frac{\text{(conc. of spiking solution (ug/ml)) x (vol. of spike (ml)}}{\text{(sample volume in ml)}}$$

11.3 Calculation of Matrix Spike Recovery.

$$\text{Percent recovery} = \frac{\text{(SSR - SR) x 100}}{\text{SA}}$$

where SSR = Spiked sample result
SR = Sample result
SA = Spike added

11.4 Calculation of Calibration Check Standard (CCS) recovery.

$$\text{Percent recovery} = \frac{\text{CCS result x 100}}{\text{True Value}}$$

11.5 Calculation of Relative Percent Difference (RPD).

$$\text{RPD} = \frac{\text{(Sample result - Duplicate result)}}{\text{\{(Sample result + Duplicate result)/2\}}} \times 100$$

12.0 QC REQUIREMENTS

12.1 Analyze a new 5 point calibration curve, at a minimum, on each analysis day. Up to eight calibration standards may be employed. The calibration curve is required to have a correlation coefficient greater than or equal to 0.995 percent. (Note: The 2.0 mg/L standard may be dropped from the curve to increase linearity.)

$$r = \frac{\Sigma(x - \bar{x})(y - \bar{y})}{\sqrt{\Sigma(x - \bar{x})^2 \Sigma(y - \bar{y})^2}}$$

Where r = correlation coefficient
x = amount of analyte
y = response of instrument

$\bar{x}$ = average of x values

$\bar{y}$ = average of y values

12.1.1 Calculate the relative error in the calibration. The criteria for this method is +/- 30%. The calibration must be reanalyzed if the criteria is not met.

12.1.1.1 Calibrations evaluated using an average response factor, the determination of the relative standard deviation (RSD) is the measure of the relative error.

12.1.1.2 Calibrations evaluated using correlation coefficient or coefficient of determination, the laboratory must evaluate relative error by either Relative Error (%RE) or Relative Standard Error(%RSE).

12.1.1.2.1 Relative error is calculated using the following equation

$$\%\,Relative\,Error = \frac{x'_i - x_i}{x_i} \times 100$$

x_i = True value for the calibration standard
x'_i = Measured concentration of the calibration standard

This calculation shall be performed for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level.he Relative Error at both of these levels shall meet the criteria specified in the method.

12.1.1.2.2 Relative Standard Error is calculated using the following equation

$$\%\,RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x'_i - x_i}{x_i}\right]^2 / (n - p)}$$

x_i = True value of the calibration level i
x'_i = Measured concentration of calibration level i
p = Number of terms in the fitting equation (average = 1, linear = 2, quadratic = 3)
n = Number of calibration points

12.2 Initially analyze all samples undiluted. If the sample concentration is higher than the highest standard, then reanalyze the sample as a dilution. Prepare the dilution so that the sample is in the mid-range of the calibration curve.

12.3 One preparation blank is required for each set of 20 samples or less or with each batch. The preparation blank is required to contain all the reagents in the same volumes as used in the preparation of the samples. The preparation blank is required to be less than the RL. If it exceeds this limit do not proceed. Check with the laboratory supervisor or manager for further directions.

12.4 After every 10 samples, every 20 readings (10 samples plus 10 background correction points) and at the end of the sample analysis sequence a digested calibration check and a reagent blank must be analyzed. Prepare the mid-point calibration check from a different stock than the calibration curve. This calibration check standard must also be

analyzed at the beginning of the analysis immediately after the calibration curve. The acceptance criterion for the mid-range calibration check standard is 90 – 110% of the true value of the standard. If they are outside of this range, do not proceed. Check with the laboratory supervisor of manager for further directions.

12.5 Analyze a reagent blank after each mid-calibration check standard. The reagent blank is required to be less than the RL. If it exceeds this limit do not proceed. Check with the laboratory supervisor or manager for further directions.

12.6 Prepare and analyze a duplicate sample for each set of 20 samples of a similar matrix or with each batch, whichever is smaller. Acceptance criteria of 20 percent relative percent difference is applied if the original and duplicate sample values are greater than or equal to 0.050 mg/l.

12.7 Prepare and analyze a matrix spike for each set of 20 samples of a similar matrix or with each batch, whichever is smaller. An acceptance criterion of 85 to 115 percent recovery is applied if the spike amount is greater than one fourth of the sample amount. If the matrix spike does not meet these criteria, then analyze the sample at a 1:5 dilution and a pH adjusted spike must be analyzed.

12.8 A spike blank or lab control must be prepared and analyzed for each set of 20 samples of a similar matrix or with each batch, whichever is smaller. The spike blank or lab control can be prepared using either soluble or insoluble hexavalent chromium as the spike. The acceptance range for the spike blank is 90 to 110 percent recovery. If the spike blank is not within that range, do not proceed. Check with the laboratory supervisor or manager for further directions.

13 DOCUMENTATION REQUIREMENTS

13.1 The analyst must document all relevant information, including all sample weights and volumes, digestion times and temperatures, all intermediate and final pH values, all times relevant to the pH adjustment process, all sample and background analysis results, and any relevant comments for any section of the digestion or analysis. Sample digestion and analysis sheets are provided.

13.2 For the analysis, all reagent identification numbers must be recorded on the sample worksheets. In addition, all reagent information such as lot numbers must also be recorded in the reagent logbook.

14 DATA REVIEW AND REPORTING

14.1 All samples must be updated to GP and GN batches in the LIMS system. The analyst must calculate all matrix spike, duplicate, external, and CCV recoveries and review the results of all blanks. These calculations may be automated or manual.

14.2 All documentation must be completed, including reagent references and spike amounts and spiking solution references.

14.3 A data file must be exported to the LIMS system and the spike amounts must be entered into the file at the GNAPP process step.

14.4 A final data package, consisting of the prep and analysis raw data, the LIMS cover page, the reagent reference pages, and the QC summary pages must be turned into the area supervisor or other senior reviewer for review.

14.5 After review by the supervisor, the data is released in the LIMS for access to the clients.

14.6 The department manager does an additional periodic review on the sample data as appropriate

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes

15.2.2 Hazardous aqueous wastes

15.2.3 Chlorinated organic solvents

15.2.4 Non-chlorinated organic solvents

15.2.5 Hazardous solid wastes

15.2.6 Non-hazardous solid wastes

16.0 ADDITIONAL REFERENCES

16.1 Refer to the spectrophotometer instrument manual(s) for additional information.

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
All	Updated to most current format
FN	Changed file name from „DAYT-WET-0333-14-SOPT-7196 AQ“

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
13	01/31/2020	Hemex Patel / Maria Ruschke
14	07/09/2021	Angela Lattanzio

END OF DOCUMENT

LAB SUPERVISOR: Natasha Vun

QA OFFICER: Olga L. Galan

EFFECTIVE DATE: 2-8-24

TITLE: COLD VAPOR ANALYSIS OF MERCURY FOR WATER SAMPLES
REFERENCES: EPA 245.1, revision 3.0 (1994); SW846 7470A, revision 1 (1994); SW846 7000B, February 2007, Revision 2.
REVISED SECTIONS: references, 8.3.1, 10.4, 11.2.1, 12.3, 12.8.2.1, 12.10.1, 12.10.2
DELETED SECTIONS: 8.4, 12.10.1.1

1.0 SCOPE AND APPLICATION

1.1 Method 7470 is a cold-vapor atomic absorption procedure approved for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. All samples must be subjected to an appropriate dissolution step prior to analysis.

1.2 Method 245.1 measures total mercury (organic + inorganic) in drinking, surface, ground, sea, brackish waters, industrial and domestic wastewater.

2.0 SUMMARY

2.1 Cold vapor mercury is a flameless AA procedure based on the absorption of radiation at 253.7 by mercury vapor. Organic mercury compounds are oxidized, and the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Results are quantitated by comparison to a daily calibration curve.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve. Detected concentrations below this concentration cannot be reported without qualification. The reporting limit for mercury water samples based on the procedures outlined in this SOP is 0.0002 mg/l.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B, Revision 2 and lab SOP EQA075. This value represents the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed, and which are processed as a unit. For QC purposes, if the number of

samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD: The calibration check standard is a mid-range calibration standard. The calibration check standard must be run at a frequency of 10 percent or less. The mid-level calibration check standard criteria are + 10 percent of the true value.

EXTERNAL CHECK STANDARD: The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. The laboratory must initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. In house limits must also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses).

SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. Assess laboratory performance against the control limits specified in the SOP. In house limits must also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample must be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses).

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 per batch. For EPA 245.1 every 10 samples and for SW846 7470A one every 20 samples needed. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results must be flagged with the appropriate footnote.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

$$\frac{(|\text{MS Result} - \text{MSD Result}|) \times 100}{} = \text{MSD RPD}$$

(MS Result + MSD Result)/2

METHOD BLANK: The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. A new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDL): This value represents the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards must be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible

for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 After the mercury digestate is reduced to Hg vapor, it must be handled in a closed system or in a hood to prevent inhalation of the toxic vapor. Make sure that the Hg instrument is vented directly to a hood.

6.0 PRESERVATION AND HOLDING TIME

6.1 All water samples must be preserved by acidification with nitric acid to a pH of 2 or lower and stored in a polyethylene or glass container.

6.1.1 If samples cannot be preserved at collection, they may be acid preserved upon receipt in the laboratory. Following acidification, the sample needs to be mixed, held for 24 hours, and then verified to be pH <2 just prior withdrawing an aliquot for processing. If for some reason such as high alkalinity the sample pH is verified to be >2, more acid must be added, and the sample held for additional 24 hours until verified to be pH <2.

6.2 All samples must be analyzed within 28 days of the date of collection.

7.0 INTERFERENCES

7.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations of sulfide (as sodium sulfide) as high as 20 mg/l do not interfere with mercury recoveries when following this method. High copper concentrations (> 10 mg/l) may also interfere with mercury recoveries.

7.2 Samples that are high in chloride such as seawater, brine, and industrial effluent may require as much as 25 ml of additional permanganate. NOTE: When chloride concentrations are high, hydroxylamine sulfate and stannous sulfate must be used in place of the corresponding chlorides.

7.3 Finally, certain volatile organic materials will also absorb at this wavelength and can interfere. It can be determined if this type of interference is present by doing a preliminary run without reagents.

8.0 APPARATUS

8.1 Leeman Hydra II automated analyzers. Refer to the instrument manuals for further details on this instrumentation, including proper venting and safety requirements. Instrument maintenance is outlined below.

8.1.1 Change the sample tubing as needed.

8.1.2 Change the drying tubing as needed.

8.1.3 Clean the exterior of the instrument as needed.

8.1.4 Adjust the Hg lamp as needed. This can be done in the software on both instruments.

8.1.5 Complete any other maintenance required to maintain the instrument in good running order including, but not limited to, cleaning the cell, changing other tubing, changing the Hg lamp, etc.

8.2 Heating Equipment.

8.2.1 Graphite heating block capable of heating at 95 °C (90-95 °C) for 2 hours.

8.3 Digestion Bottles. Disposable plastic digestion tubes are used with the graphite heating block.

8.3.1 Disposable plastic digestion tubes (65 ml volume) with tops for graphite heating block (Certificate of Analysis is kept on file with the lab).

8.4 Automatic pipettor bottles. Refer to EQA063 for calibration information.

9.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. Deionized water must be used whenever water is required. All solutions listed below may be scaled up or down proportionally as needed.

9.1 Sulfuric acid, concentrated.

9.2 Nitric acid, concentrated. This acid must have a low mercury content.

9.3 Dilution acid. To approximately 400 ml of DI water, add 33.4 ml of concentrated sulfuric and 16.6 ml of concentrated nitric. Dilute to a final volume of 1000 ml. This dilution acid is used for making dilutions of digested samples.

9.4 Stannous chloride. Add 25 ml of concentrated hydrochloric acid to approximately 400 ml of DI water. Dilute to 500 ml with DI water and mix well. Add 50 g of stannous chloride dihydrate or 42 g of stannous chloride anhydrous and dissolve. Make sure that this solution is dissolved while in use.

9.4.1 Stannous sulfate may be used in place of stannous chloride.

9.5 Sodium chloride-Hydroxylamine hydrochloride. Add 240 g of sodium chloride and 240 g of hydroxylamine hydrochloride to 2000 ml of water. Mix well. Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

9.6 Potassium Permanganate, 5 percent solution, w/v. Add 50 g of potassium permanganate to 1000 ml of water and mix well. **Caution** - Potassium permanganate is a strong oxidizing agent. Handle with care.

9.7 Potassium Persulfate, 5 percent solution, w/v. Dissolve 50.0 g of potassium persulfate in 1000 ml of water and mix well. **Caution** - Potassium persulfate is a strong oxidizing agent. Handle with care.

9.8 Mercury standard solutions

9.8.1 10ppm Hg solution. Using a 1.00 ml volumetric pipet or autopipet, add 1.00 ml of 1000 ppm stock to a 100 ml volumetric flask containing approximately 75 ml of water and 2.0 ml of concentrated nitric acid. Dilute to volume with water and mix well. This standard may be held for up to 28 days.
1000 ppm Stock can be purchased from the outside vendor such as Ultra and Inorganic ventures, they are light sensitive and must be kept away from the direct light.

9.8.1.1 The 10ppm external source must be made up following the directions in 9.8.1. External stock solution must be from different vendor than calibration source.

9.8.2 **30.0**ppb Hg solution. Using an autopipet, add **0.300** ml of 10 ppm Hg solution to a 100 ml volumetric flask containing approximately 75 ml of water and 2.0 ml of concentrated nitric acid. Dilute to volume with deionized water and mix well. This standard must be made fresh daily.

9.8.2.1 The 30.0 ppb external source must be made up following the directions in 9.8.2.

9.8.3 **3**ppb Hg solution. Using volumetric pipets or autopipets, add 10.0 ml of **30.0** ppb Hg solution to a 100 ml volumetric flask containing approximately 75 ml of DI water and 2.0 ml of concentrated nitric acid. Dilute to volume with deionized water and mix well. This standard must be made fresh daily.

10.0 WATER DIGESTION FOR GRAPHITE HEATING BLOCK

Below is a step-by-step procedure for the digestion and analysis of water samples for mercury.

10.1 If necessary, acid rinse disposable digestion tubes with 10% nitric acid and deionized water before use.

10.2 Make up a standard curve consisting of 5 standards and a blank. Suggested concentrations are shown below. All standards are made up to a final volume of **30** ml. Different concentrations may also be used, as long as all of the method requirements are met. Make sure to clearly label each bottle. Calibration standards must be prepared fresh with each digestion batch.

ml of 3 ppb Hg solution	ml of 30 ppb Hg solution	Ml of DI water	Total ug of Hg	ug/L of Hg
0.000	0.000	**30**	0.000	0.000
2.00	0.000	**28.0**	**0.006**	0.20
5.00	0.000	**25.0**	**0.015**	0.50
0.00	1.00	**29.0**	**0.030**	1.00
0.00	2.50	**27.5**	**0.075**	2.50
0.00	5.00	**25.0**	**0.150**	5.00

10.3 Before starting the analysis, check the initial pH of the sample with pH paper and verify that it is < 2. Below is procedure for the checking pH and preservation of metals samples when pH

is NOT <2. Add a small amount of concentrated nitric acid (~2mls) dropwise to the sample and mix. Using a pipet tip, place a small amount of sample on a piece of pH paper or pour directly on the pH paper at the sink to confirm that the pH is <2. Document the date and time of the pH adjustment and the lot number of the nitric acid used for the preservation on the preservation log. Mark the date and time of preservation on the bottle to ensure that the analysis is not started for 24 hours from the time of preservation.

10.3.1 For highly basic or buffered samples, where more than a few drops of acid are required, adjust an aliquot of the sample rather than the whole amount and record the amount of acid used for the adjustment.

10.3.2 For each sample, homogenize the sample well and pour out a representative aliquot of the sample into the digestion tube to the 30 ml mark. A smaller volume may be used if there are matrix problems or known high levels of mercury in the sample. Smaller than 5 ml volume must be taken using calibrated auto pipet. Digestion tubes are calibrated at all graduation lines by the vendor, confirm the provided certificate of analysis.

10.3.3 Samples for TCLP matrix or leachate spikes will be received unpreserved from the TCLP leaching area. These must be aliquoted, spiked, and preserved on receipt in the metals prep area. For handling leachate oil samples refer to the SOP EMA228.

10.4 Make up additional quality control samples as shown below, using a final volume of **30** ml for each check standard. (Note: if a different standard curve is run, then the levels of the CCV and ICV standards must be adjusted accordingly in accordance with the requirements in the methods) Make sure to clearly label each bottle. Make sure to prepare enough CCV checks for the entire run. The ICV check must be from an alternate source of standards than the calibration curve. The CCV must be made from the same source as the calibration curve. A low check standard at the level of the CRDL (0.20 ug/l) is also required. This 0.20 ug/l check can be made up as outlined for the standard curve. Witness signature is required in the reagent sheet or prep log for pouring, aliquoting and spiking samples.

Sample ID	**ml of** 30 **ppb Hg solution**	**ml of DI water**	**Total ug of Hg**
CCV Check(s)	2.5	27.5	**0.075**
MB	0.0	30	**0.0**
MS	2.0	(a)	**0.06 (b)**
MSD	2.0	(a)	**0.06 (b)**
ICV	3.0	**27.0**	**0.09**
LCS(BS)	2.0	**28.0**	**0.06**
Post-spike	1.0	14 ml sample	**0.06 (b)**

(a) **30** ml of sample
(b) plus, the level of Hg in the sample.

10.5 To all samples, QC, and standards add the reagents listed below, swirling the samples well after each addition of reagent. Allow the samples to stand for at least 15 minutes after the addition of the permanganate. If the sample decolorizes, add additional permanganate until the purple color persists. When additional potassium permanganate added to a sample, equal amounts must be added to the associated batch quality control samples such as

Method blank, Blank spike, Matrix spike, Matrix duplicate, QC sample and Duplicate.

- **1.5** ml of conc. sulfuric acid.
- **0.75** ml of conc. nitric acid.
- **4.5** ml of 5% permanganate solution.

Wait 15 minutes, then:

- **2.4** ml of potassium persulfate solution.

10.5.1 All of the additions shown can be done with a bottle pipettor which must be accurate to within a range of 90 to 110%.

10.6 Cap the samples and place them in the graphite heating block and heat for 2 hours at 90 to 95 °C. Record the digestion times and temperature.

Note: For method 245.1 Calibration standards, Continuing calibration standards, initial calibration verification and continuing calibration blanks are not digested.

10.7 Enter the prep data into the LIMS system, double checking all volumes and spike amounts. After the prep data is checked, it can be approved and is available for use in the final calculations.

11.0 COLD VAPOR ANALYSIS PROCEDURE HYDRA II

11.1 While the samples are digesting, begin setting up the Leeman analyzer following the steps outlined below. Additional instructions are available in the instrument operators' manual.

11.1.1 Turn on the nitrogen and adjust to 60 to 90 psi. Turn on the instrument power if it is not already on.

11.1.2 Check the pump tubing and make sure that it is not flattened. Change if appropriate. Put the tubing in the clamps on the pump. Check the drying line and make sure that it is clean. Put fresh stannous chloride solution in the stannous chloride bottle. Fill the rinse bath or rinse bottle with fresh 10% nitric acid. The bath must be filled no more than ¾ full. Place the autosampler line and the stannous chloride line in the rinse container.

11.1.3 Turn on the analyzer and allow it to warm up.

11.1.3.1 For the Hydra AA II, open the Envoy software. Go to Method and click Instrument Control. On the Instrument Control page, click the startup icon. This will turn on the lamp, gas, and pump. You may also turn on/off the lamp, gas and pump individually on the Instrument Control Page.

11.1.4 Tighten the pump clamps until the flow is coming evenly through the lines. Do not overtighten.

11.1.4.1 Go to the Instrument control tab and pick the gas control test option. The input

must be approximately 0.25 LPM. If the pressures are not correct, check with the area supervisor or manager before proceeding.

11.1.5 Start a batch to save your data.

11.1.5.1 Create a new chapter (Data File) by clicking Analysis. The batch must normally be named H5 followed by the month date and year, followed by the matrix designation for the batch, following by the run number. For example, the first water batch on instrument for 3/24/03 would be named H5032403w1. The realtime print option can also be turned on from this tab.

11.1.5.2 Set up autosampler racks containing the samples that are going to be run.

11.1.5.3 Create a new sequence by clicking sequence--new. Type the sequence name. After typing the samples in to sequence page make sure to click update and save. CCV and CCB checks can be entered in the macro column of the sequence page.

11.1.6 Set up the calibration.

11.1.6.1 Go to the Method menu, enter or verify the standard concentration by clicking on the standard tab. Also select number of replicates to be run for each standard. Normally one replicate is run per standard. The check standard concentrations and acceptance ranges are also defined under this standard info tab. Make sure to always click apply when any changes are made in a tab.

11.2 Add hydroxylamine hydrochloride to all samples and standards as outlined below.

11.2.1 Add **1.8** ml of hydroxylamine hydrochloride solution to each standard and sample and swirl until the solution has been completely decolorized. Dilute to a final volume of **50** ml and swirl to mix.

11.2.2 The hydroxylamine hydrochloride can be added using a bottle pipettor which is accurate in a range of 90 to 110%.

11.3 Measure out aliquots of the digested standards and samples into the autosampler cups. Work from the prep log and double check all transfers. Let all samples sit uncovered in the open autosampler vials for a minimum of one minute. Place the racks in the autosampler. Move the stannous chloride line into the stannous chloride bottle.

11.4 Start the calibration.

12.4.1 Click run sequence. The instrument will run the calibration and then pause. Click stop. Go to the Calibration page. Accept the calibration and then print the calibration. Click the Document icon, then choose HG5-PDF. Rename the file as MA*****_cal.

11.5 After the calibration has been accepted, start to run the samples.

12.5.1 For the Hydra AA II, go to the Sequence page. Right click on the first sample (ie. ICV) and click start from here.

11.6 Review the data. Any samples that are over the range of the curve must be diluted with the dilution acid (see Section 9.3) and reanalyzed. It is recommended that any sample analyzed after a sample with a value over the curve be reanalyzed for confirmation. Make sure to bracket every 10 samples with CCV and CCB checks.

11.7 Both paper and electronic reports can be generated using the report option. Never delete any samples from the reports. Electronic reports must be transferred into the LIMS system where the final calculations are done.

11.7.1 Go to analysis-Click result-Click chapter. Then go to report and select report spec. The normal report spec is "ACCUTEST". Click OK. Click on chapter in order to select all samples. Then click report output and then csv.file. Save as MA*****.csv. To print, select printer output and then type the report title (i.e. MA*****) and enter OK.

11.8 The calculations are done in the LIMS as described below. A final volume of 30.0 ml is used for calculation purposes for graphite heating block digestions. (The volume of 50.0 ml is factored out since all standards and samples are brought up to the same final volume and standard concentrations are calculated based on 30.0 ml.)

Final sample concentration in mg/L =

$$\frac{\text{concentration in the digestate in ug/l x final volume in ml}}{\text{Initial volume in ml}}$$

11.9 Review the data in the LIMS, adding comments and accepting results as appropriate.

11.10 Shut down the instruments.

11.10.1 To shut down the Hydra AA II, move the stannous chloride line from the stannous chloride bottle to the 10% HNO_3 rinse bottle. Let the system rinse with 10% HNO_3 for several minutes. Then switch the line to DI water bottle and let rinse for several more minutes. Let the pump and gas run until the lines are completely dry. Then go to instrument control menu and click off icon for Lamp, Gas and Pump.

12.0 QUALITY CONTROL

Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

12.1 Instrument Detection Limits (IDLs). The instrument detection limits must be done a minimum of once per year or when instrument conditions change significantly. The IDL is generated by running 10 replicates of a digested blank. The IDL is then defined as 3 times the standard deviation of the 10 replicates of the blank.

12.2 Method Detection Limits (MDLs). MDLs are determined using the procedure specified in 40 CFR, Part 136, Appendix B, Revision 2, and lab SOP EQA075.

12.3 Instrument Calibration. The instrument must be calibrated daily or at a minimum of once every 24 hours and each time the instrument is set up. Calibration standards must be prepared fresh with each preparation batch. A minimum of a blank and 5 standards are required. The

correlation coefficient of the curve must be a minimum of 0.995. The Relative Error percent (RE%) must be calculated for low and mid points of the curve. The Relative Error for the midpoint is 10% and for low point is 30%. These criteria must be documented and kept on file. No samples must be analyzed until all of the calibration criteria are met. Resloping is acceptable as long as it is immediately preceded and immediately followed by a complaint CCV and CCB.
NOTE: method 245.1 calibration standards must not be digested.

12.4 Linear Dynamic Range (LDR). For each instrument, the upper limit of the linear dynamic range must be established. A linear calibration must be prepared from 3 standards, one of which is close to the upper limit of the linear range. The LDR is determined by analyzing succeedingly higher standard concentrations of mercury until the observed analyte concentration is no more than 10 percent below the true value of the standard. Sample concentrations that are greater than 90% of the determined upper LDR limit must be analyzed using dilutions. The LDR must be verified annually or whenever there is a significant change in the instrument's analytical performance.

12.5 Quality Control Sample (also referred to as Initial Calibration Verification Standard (ICV)). At a minimum of once per quarter, a standard from a different source than the calibration standard must be analyzed. Normally this is analyzed at the beginning of the run after the CCV and CCB checks. The ICV must be within 10 percent of the true value. It is recommended that this standard be analyzed with each run so that it is included with all client reports. For SW846 7470A, this standard must be at a concentration near the midpoint of the calibration curve. If the ICV is outside of the acceptance limits, then the problem must be corrected and the ICV reanalyzed and shown to be within QC limits before any samples can be reported. All reported samples must be bracketed by an ICV which meets acceptance criteria.

12.5.1 If the ICV is biased high and all sample results are < RL, then, at the discretion of the data reviewer, data may be reported.

12.6 Method Blank. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. The method blank must contain mercury at < 2.2 times the MDL or < ½ the RL, whichever is less. If the method blank value is over that limit, the samples must be redigested or reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit. (Note: Not all program codes allow this. Verify prior to proceeding.)

12.6.1 Note: For practical purposes, reviewing the method blank data to ½ RL will meet the 2.2 times MDL requirement.

12.7 Lab Control Sample. The laboratory must digest and analyze a laboratory control sample (spike blank) with each set of samples. A minimum of one lab control sample is required for every 20 samples. For drinking water samples, a low-level spike blank (at RL level) is required for each different digestion day when drinking water samples are analyzed. The low-level BS must be within 50-150% recovery. For method 245.1, the laboratory must assess laboratory performance of an aqueous lab control against recovery limits of 85 to 115 percent. For method 7470A, the laboratory must assess laboratory performance of an aqueous lab control against

recovery limits of 80 to 120 percent. In either case, if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit, then the sample results can be reported with no flag. If the lab control recovery is low or there are samples above the reporting limit, then all affected samples must be re-digested and reanalyzed.

12.8 Matrix Spike.

12.8.1 For method 245.1, the laboratory must add a known amount of each analyte to a minimum of 1 in 10 samples. The spike recovery must be within the limits of 70 to 130. If a matrix spike is out of control, then the results must be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and must be footnoted to that effect.

12.8.2 For method 7470A, the laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The laboratory must assess the matrix spike recovery against limits of 75 to 125. (In house control limits are also generated on an annual basis and are used to support the default limits.) An exception to this rule occurs where the sample concentration exceeds the spike concentration by a factor of 4 or more.

12.8.2.1 If the matrix spike fails this criterion, then the sample from which the MS/MSD aliquots were prepared should also be spiked with a **post digestion spike**. An analyte spike is added to a portion of a prepared sample, or its dilution, and must be recovered to within 80-120% of the known value. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed.
For post-digest spike sample, add 1 ml of 30 ppb Hg solution to the 14 ml of the digested sample (see Table section 10.4).

12.8.3 Both the matrix spike amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero. Refer to the calculation shown below.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{(Amount Spiked)}} \times 100 = \text{MS Recovery}$$

12.9 Matrix Spike Duplicate or Matrix Duplicate. The laboratory must digest a matrix spike duplicate or a duplicate sample for a minimum of 1 in 20 samples. Matrix spike duplicates are normally used unless otherwise specified by client requirements. The relative percent difference (rpd) between the matrix spike duplicate and the matrix spike or between the duplicate and the sample must be assessed. The calculations for both rpds are shown below.

12.9.1 For method 245.1, the control limits for the matrix spike duplicates or the duplicates are calculated on an annual basis and are used to assess whether a matrix spike duplicate or a duplicate is in control. If it is out of control, then the results must be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of $\pm$ the reporting limit, then the duplicate is considered to be in control.

12.9.2 For method 7470A, the duplicate or matrix spike duplicate RPD must be assessed against a limit of 20% RPD. (In house control limits are also generated on an annual

basis and are used to support the default limits.) If it is out of control, then the results must be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

12.9.3 Both the duplicate amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero. Refer to the calculations shown below.

$$\frac{(\text{Sample Result} - \text{Duplicate Result}) \times 100}{(\text{Sample Result} + \text{Duplicate Result}) \times 0.5} = \%\ \text{RPD}$$

or

$$\frac{(|\text{MS Result} - \text{MSD Result}|) \times 100}{(\text{MS Result} + \text{MSD Result})/2} = \text{MSD \%RPD}$$

12.10 Continuing Calibration Verification. (Also known as the instrument performance check solution.) The CCV must be from the same source as the calibration curve.

12.10.1 Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run. If the CCV solution is not within a method specified range of the true value, then no samples can be reported in the area bracketed by that CCV. (Note: the exception is if the CCV is biased high and the samples are less than the detection limit. In that case, the samples can be reported with no flag.) The CCV concentration must be at or near the mid-range of the calibration curve. Passing criteria for CCV is ±10%.

12.10.2 For 245.1 method, the ICCV (called ICCV for LIMS identification) check must be analyzed at the beginning of the run, immediately after the instrument is calibrated. This first check must be within± 5% of the true value. If it is not and there is not a problem with the standard solution, the instrument must be recalibrated and rechecked. After that, the bracketing CCV for 245.1 method must follow the criterion section 12.10.1.

12.11 Continuing Calibration Blank. Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run. If the CCB is not less than the reporting limit, then no samples can be reported in the area bracketed by the failing CCB.

12.12 CRA (Low) Check. For all runs, a low check at the level of the CRDL (0.20 ug/l) or reporting limit must be analyzed at the beginning of the run before analyzing any samples, but not before the ICV. No specific acceptance criteria are listed in any of the methods for this standard at this time. The method criteria of 70-130% recovery is applied to this low check standard. If this criterion is not met, then all samples associated with this CRA check must be reanalyzed along with a compliant CRA check.

12.12.1 If the CRA is biased high and there is no mercury found in the samples, then the sample results may be reported for mercury. If the CRA is biased high and there is mercury found in the samples, then the samples with Hg at levels ranging from the

CCV to the high standard may be reported. Samples with levels of mercury between the CRA and the CCV standard may be biased high and cannot be reported.

12.12.2 Some client may require additional bracketing low checks to be analyzed. Client specific limits may also be required. Check with the area supervisor or manager for more information.

12.12.3 Measurement for Relative Error (%RE).
Relative error is calculated using the following equation:

$$\% \text{ Relative Error} = \frac{x'i - xi}{xi} \times 100$$

xi = True value for the standard
x'i = Measured concentration of the standard

13.0 DOCUMENTATION REQUIREMENTS

Refer to the laboratory Quality Assurance Manual for additional documentation requirements.

13.1 Sample Worksheets. Digestion data sheets for the Hg water samples must show all digestion information including the sample ID's, sample volumes, bottle numbers, start times, end times, and pressure or temperature, as appropriate for all digestions. The digestion method (i.e digestion block) must be indicated on the digestion sheet. All sample information must be clearly entered on these sheets. In addition, any unusual characteristics of the samples or the digestion procedure must be noted in the Comments sections. Make sure also that all dilutions are clearly documented.

13.2 Standards and Reagents. All stocks and reagents must be recorded in the reagent logbook. All standards must be recorded on the digestion log with the samples.

13.3 Any run comments must be written on the raw data for the analysis and on the run log in the LIMS.

13.4 Annual bottle calibration verifications must be documented in the Mercury Bottle calibration log.

14.0 DATA REVIEW AND REPORTING

14.1 All samples must be updated to QC batches in the LIMS system. The analyst is responsible for reviewing all data for compliance with the QC outlined in this SOP. They are responsible for making sure that the raw data is fully documented, and it is loaded into the LIMS system. They are responsible for submitting samples for redigestion and reanalysis, when appropriate.

14.2 After the analyst review is completed, the supervisor or a designated reviewer shall review the run for technical compliance to the SOP. The reviewer is also responsible for making sure that the QC calculations are done correctly and that appropriate flags are added.

14.3 After the reviewer completes their review, the data is released for client access in the LIMS. The raw data and the run log are submitted to the area manager. The manager periodically does an additional review on data for technical completeness. Any hardcopy raw data is transferred to the report generation department for scanning and storage. Instrument data is transferred electronically.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS 004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes.

15.2.2 Hazardous aqueous wastes.

15.2.3 Chlorinated organic solvents.

15.2.4 Non-chlorinated organic solvents.

15.2.5 Hazardous solid wastes.

15.2.6 Nonhazardous aqueous wastes.

16.0 ADDITIONAL REFERENCES

16.1 Leeman Hydra II instrument manual.

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
11.2.1	Removed reference to glass cylinder
8.3.1	Added reference to the COA for digestion tubes.
12.3	Revised requirement for RE% for calibration curve
10.4	Revised table for spiking
12.8.2.1	Added prep for PS
12.10.1	Added criterion for CCV
12.10.2	Clarified the purpose of ICCV

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed
8.4	Glass cylinders are not in use
12.10.1.1	Reference for RE% for CCV and CRA

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added
References	Added reference to EPA 7000B

History of Revisions

Version #	Date of Revision	Revised By
24	1/22/20	Olga Azarian
25	1/4/21	Rakesh Pathak
26	2/14/22	Olga Azarian
27	8/31/23	Olga Azarian
28	9/29/23	Natasha Verma
29	03/07/24	Olga Azarian

END OF DOCUMENT

LAB SUPERVISOR: Natasha Vrun

QA OFFICER: Olga G. Agaian

EFFECTIVE DATE: 3-7-2024

TITLE: COLD VAPOR ANALYSIS OF MERCURY FOR SOIL SAMPLES
REFERENCES: SW846 7471B, revision 2, February 2007; SW846 7000B, February 2007, Revision2.
REVISED SECTIONS: references,7.4, 10.6, 12.3
DELETED SECTIONS: 7.4.1, 7.4.2, 7.4.3, 7.4.4, 7.5

1.0 SCOPE AND APPLICATION

1.1 This method can be applied for the analysis of mercury in soils, sediments, bottom deposits, and sludge type materials. The reporting limit for mercury soil samples, based on a 0.6 g sample size, is 0.033 mg/kg.

2.0 SUMMARY

2.1 Cold vapor mercury is a flameless AA procedure based on the absorption of radiation at 253.7 by mercury vapor. Organic mercury compounds are oxidized, and the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Results are quantitated by comparison to a daily calibration curve.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve. Detected concentrations below this concentration cannot be reported without qualification.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B Revision 2, and SOP EQA075.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed, and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD. The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard be run at a frequency of approximately 10 percent. Recovery requirements vary by method. For this method a recovery from 90 to 110% is required. (For some methods this is mandatory and for some it is a recommendation only. Refer to individual method SOP's)

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. Assess laboratory performance against the control limits specified in the SOP. If the lab control is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent must be used.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample must be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results must be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and must be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent must be used.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

$$\frac{(|\text{MS Result} - \text{MSD Result}|) \times 100}{(\text{MS Result} + \text{MSD Result})/2} = \text{MSD RPD}$$

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLS). The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLs must be determined approximately once per year for frequently analyzed parameters.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards must be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation.

LOWER LIMIT OF QUANTITATION CHECK (also referred to as CRI, CRA, or LLQC). For all runs, a low check at the level of the reporting limit must be analyzed at the beginning of the run before analyzing any samples, but not before the ICV. A method criterion of 50 to 150% recovery is applied to this low check standard. If this criterion is not met, then all samples associated with this CRA check must be reanalyzed along with a compliant CRA check.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS-Dayton Chemical Hygiene Plan Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 After the mercury digestate is reduced to Hg vapor, it must be handled in a closed system or in a hood to prevent inhalation of the toxic vapor. Make sure that the Hg instrument is vented directly to a hood.

6.0 PRESERVATION AND HOLDING TIME

6.1 All solid samples must be stored at 4 ± 2ºC until the time of digestion.

6.2 All samples must be analyzed within 28 days of the date of sampling.

7.0 APPARATUS

7.1 Leeman Hydra II AA automated analyzers. Refer to the instrument manuals for further details on this instrumentation, including proper venting and safety requirements. Instrument maintenance is outlined below.

7.1.1 Change the sample tubing as needed.

7.1.2 Change the drying tubing as needed.

7.1.3 Clean the exterior of the instrument as needed.

7.1.4 Adjust the Hg lamp as needed. This can be done in the software on both instruments.

7.1.5 Complete any other maintenance required to maintain the instrument in good running order including, but not limited to, cleaning the cell, changing other tubing, changing the Hg lamp, etc.

7.2 Graphite heating block. Capable of heating at 95 ± 3 °C for 2 hours.

7.3 Digestion Bottles. Disposable plastic digestion tubes (65 ml volume) with tops for graphite heating block.

7.4 Digestion tubes

7.5 Analytical Balance, 4 places. Calibration must be verified daily before use with NIST traceable weights.

7.6 Automatic pipettor bottles. The calibration on these bottles must be verified as outlined in EQA063.

7.7 Volumetric pipets, class A.

7.8 Glass digestion tubes. The purchased glass tubes have 50ml and 100 ml marks and are calibrated for these two volumes by the manufacturer. Calibration certificate is provided by manufacturer. This can be used for digestion instead of disposable digestion tubes.

7.9 Disposable Wood Spatulas

7.10 Ceramic Mixing Bowl

8.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. Deionized water must be used whenever water is required. All solutions listed below may be scaled up or down proportionally as needed. Different reagents are required for the different heating techniques.

8.1 Digestion Block Reagents.

8.1.1 Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated nitric.

8.1.1.1 Make sure to only prepare the amount of acid that will be needed for the prep and analysis.

8.1.1.2 This preparation must be done in a hood.

8.1.2 Dilution Acid: To approximately 800 ml of DI water, add 37.5 ml of concentrated HCl and 12.5 ml of concentrated nitric acid. Dilute to 1000 ml with DI water and mix well. This dilution acid is used for making dilutions of digested samples from the digestion block digestion procedures.

8.1.3 Potassium permanganate, 5% solution: Dissolve 50 g of potassium permanganate in 1000 ml of DI water. **Caution** - Potassium permanganate is a strong oxidizing agent. Handle with care.

8.1.4 Stannous chloride. Add 7.5 ml of concentrated sulfuric acid to approximately 400 ml of DI water. Dilute to 500 ml with DI water and mix well. Add 50 g of stannous chloride di-hydrate or 42 g stannous chloride anhydrous and dissolve. Make sure that this solution is dissolved while in use.

8.1.4.1 Stannous sulfate may be used in place of stannous chloride.

8.1.4.2 If clogging occurs during analysis using the automated analyzer, then a less concentrated solution may be used.

8.1.5 Sodium chloride-Hydroxylamine hydrochloride or Sodium Chloride-Hydroxylamine hydrosulfate. Add 240 g of sodium chloride and 240 g of hydroxylamine hydrochloride to 2000 ml of water. Mix well. Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

8.2 Mercury standard solutions.

8.2.1 10 ppm Hg solution. Using a 1.00 ml volumetric pipette, add 1.00 ml of 1000 ppm stock to a 100 ml volumetric flask containing approximately 75 ml of water and 2.0 ml of concentrated nitric acid. Dilute to volume with water and mix well. This standard may be held for up to 28 days.
1000 ppm Stock can be purchased from the outside vendor such as Ultra and Inorganic ventures, they are light sensitive and must be kept away from the direct light.

8.2.1.1 The 10-ppm external source must be made up using a different mercury stock and following the directions in 8.2.1. External stock solution must be from different vendor than calibration source.

8.2.2 100 ppb Hg solution. Using a 1.00 ml volumetric pipette, add 1.00 ml of 10 ppm Hg solution to a 100 ml volumetric flask containing approximately 75 ml of water and 2.0 ml of concentrated nitric acid. Dilute to volume with deionized water and mix well. This standard must be made fresh daily.

8.2.2.1 The 100-ppb external source must be made up following the directions in 8.2.2.

8.2.3 10 ppb Hg solution. Using a 10.0 ml volumetric pipette, add 10.0 ml of 100 ppb Hg solution to a 100 ml volumetric flask containing approximately 75 ml of DI water and 2.0 ml of concentrated nitric acid. Dilute to volume with deionized water and mix well. This standard must be made fresh daily.

8.2.4 Teflon Chips

8.2.5 Solid Lab Control (Mixture of Metals analytes in Soil Matrix).This is commercially purchased.

9.0 INTERFERENCES

9.1 Potassium permanganate is added to eliminate possible sulfide interferences. Concentrations as high as 20 mg/kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water. High copper concentrations (> 10 mg/kg) may also interfere with mercury recoveries.

9.2 Samples that are high in chlorides may require additional permanganate because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 254 nm.

Care must be taken to assure that free chlorine is absent before the mercury is reduced and analyzed. This may be accomplished by using an excess of the hydroxylamine hydrosulfate.

9.3 Certain volatile organics may also absorb at this wavelength and can interfere.

10.0 GRAPHITE DIGESTION BLOCK PROCEDURE FOR SOIL DIGESTION

10.1 Make up a standard curve consisting of 5 standards and a blank. Suggested concentrations are shown below. Different concentrations may also be used, as long as all of the method requirements are met. Make sure to clearly label each digestion tube. Calibration standards must be prepared fresh each day. Add 5 ml of DI water to each standard before digestion. The final concentration of Hg is calculated in the final digestate.

Ml of 10 ppb Hg solution	ml of 100 ppb Hg solution	Total ug of Hg	ug/L of Hg
0.000	0.000	0.000	0.000
2.00	0.000	0.020	0.20
5.00	0.000	0.050	0.50
0.00	1.00	0.100	1.00
0.00	2.50	0.250	2.50
0.00	5.00	0.500	5.00

10.2 Samples. For each sample, homogenize the sample well, weigh out an amount of wet sample equivalent to 0.5-0.6 g of dry sample into a numbered digestion tubes, Label each digestion tube. The sample must be weighed out using an analytical balance and the weights must be recorded. Make sure that the sample identification is accurately recorded with the digestion tube numbers on the sample digestion log. A solid lab control must be prepared in the same manner.

10.2.1 Make sure that the sample has been thoroughly mixed before weighing out the representative sample. Discard rocks, sticks, etc. from the sample. (Refer to the SOP EQA042 for proper sample aliquot procedures). All homogenization and sample handling must be done with wooden spatulas and ceramic (or other non-metal) bowls.

10.2.2 If the sample is leachate oil, measure 0.6ml of the sample and follow the steps starting at section 10.7. Leachate oil samples are handled using soil procedure on a volume basis.

10.2.3 If the sample is a wipe, weighing is not necessary. Transfer the entire wipe into the labeled digestion tube and proceed with the digestion following steps 10.7. Extra wipes must be supplied by the client for the matrix spike or duplicate.

10.3 In addition to the samples, a spike blank and a method blank must be set up with each batch of 20 samples or less. A solid lab control sample is used in addition to the blank spike by client requirement. A matrix spike, a matrix spike duplicate or a duplicate must be set up with each batch of 20 samples. Matrix spike duplicates are normally used unless otherwise specified by client requirements.

10.3.1 For leachate oil QC samples use 0.6ml of leachate and proceed with section 10.7.

10.4 For the method blank and spike blank, instead of weighing out soil add approximately 0.6 g of Teflon chips to the digestion tube. Add the spiking solution to the blank spike after the chips are in the tube.

10.5 Solid lab control must be weighed out in the same manner as a sample, but weight can be adjusted based on the concentration. See area supervisor for the weight information.

10.6 Make up additional quality control samples as shown below. (Note: if a different standard curve is run, then the levels of the CCV and ICV standards must be adjusted accordingly in accordance with the requirements in the method.) Make sure to clearly label each digestion tube. Make sure to prepare enough CCV checks for the entire run. The ICV check must be from an alternate source of standards than the calibration curve and at a different level than the CCV or the calibration standards. A low check standard is also required. This 0.20 ug/l check can be made up as outlined for the standard curve.

Sample ID	ml of 100 ppb Hg solution	ml of DI water	µg/l of Hg
CCV Check(s)	2.5	97.5	2.5
MB	0.0	100	0.0
BS	2.0	98	2.0
MS	2.0	98	2.0
MSD	2.0	98	2.0
Spike blank	2.0	98	2.0
Duplicate*	0.0	0.0	0.0
ICV	3.0	97	3.0
Post-spike	1.0	49 sample	2.0

*Per project specification.

10.7 To all samples, QC, and standards add 5 ml of DI water and 5 ml of aqua regia and then heat for 2 minutes in a digestion block set at 95 ± 3 °C. These reagents can be added with a bottle pipettor that is accurate to within 90 to 110%.

10.8 Cool the samples and then add 25 ml of DI water and 15 ml of potassium permanganate solution to each sample and mix thoroughly. Allow the samples to stand for at least 15 minutes after the addition of the permanganate. If the sample decolorizes, add additional permanganate until the purple color persists. When additional potassium permanganate added to a sample, equal amounts must be added to the associated batch quality control samples such as Method blank, Blank spike, Matrix spike, Matrix duplicate, QC sample and Duplicate.

10.8.1 These reagents can be added with a bottle pipettor that is accurate to within 90 to 110%.

10.8.2 For samples containing a mixture of solvent and water, take a sample aliquot of approximately 20 g (lower for higher solvent samples) of sample and add the reagents listed above. Pour the sample in a beaker and heat on a hot plate at 95 ± 3 °C until no solvent layer is visible. Then transfer the digestate to a digestion tube and proceed as outlined below. A method blank and spike blank must also be taken through this entire procedure.

10.9 Cap the samples and place them in the graphite digestion block for 30 minutes at 95 ± 3 °C. Record the temperature and time for each digestion batch on the analysis sheet. Remove and cool.

10.10 Enter the prep data into the LIMS system, double checking all weights and spike amounts. After the prep data is checked, it can be approved and is available for use in the final calculation.

11.0 MERCURY ANALYSIS PROCEDURE HYDRA AA II

11.1 While the samples are digesting, begin setting up the Leeman analyzer following the steps outlined below. Additional instructions are available in the instrument operator's manual.

11.1.1 Turn on the nitrogen and adjust to 60 to 90 psi. Turn on the instrument power if it is not already on.

11.1.2 Check the pump tubing and make sure that it is not flattened. Change if appropriate. Put the tubing in the clamps on the pump. Check the drying line and make sure that it is clean. Put fresh stannous chloride solution in the stannous chloride bottle. Fill the rinse bath or rinse bottle with fresh 10% nitric acid. The bath must be filled no more than ¾ full. Place the autosampler line and the stannous chloride line in the rinse container.

11.1.3 Turn on the analyzer and allow it to warm up.

11.1.3.1 Open the Envoy software. Go to Method and click Instrument Control. On the Instrument Control page, click the startup icon. This will turn on the lamp, gas, and pump. You may also turn on/off the lamp, gas and pump individually on the Instrument Control Page.

11.1.4 Tighten the pump clamps until the flow is coming evenly through the lines. Do not over tighten.

11.1.5 Go to the Instrument control tab and pick the gas control test option. The input must be approximately 0.25 LPM. If the pressures are not correct, check with the area supervisor or manager before proceeding.

11.1.6 Start a batch to save your data.

11.1.6.1 Create a new chapter (Data File) by clicking Analysis. The batch must normally be named H5 followed by the month date and year, followed by the matrix designation for the batch, following by the run number. For example, the first water batch on instrument for 3/24/03 would be named H5032411w1. The realtime print option can also be turned on from this tab.

11.1.7 Set up autosampler racks containing the samples that are going to be run.

11.1.7.1 Create a new sequence by clicking sequence-new. Type the sequence name. After typing the samples in to sequence page make sure to click

update and save. CCV and CCB checks can be entered in the macro column of the sequence page.

11.1.8 Set up the calibration.

11.1.8.1 For the Hydra AA II, go to the Method menu, enter or verify the standard concentration by clicking on the standard tab. Also select number of replicates to be run for each standard. Normally one replicate is run per standard. The check standard concentrations and acceptance ranges are also defined under this standard info tab. Make sure to always click apply when any changes are made in a tab.

11.2 Finish the preparation of the samples and standards as outlined below.

11.2.1 For samples that were digested in the water bath or the graphite digestion block, add 6 ml of hydroxylamine hydrochloride or hydroxylamine hydrosulfate to each sample and standard and mix well. Transfer the entire digestate to a calibrated glass tube. Rinse the digestion tube 3 times with approximately 10 ml aliquots of DI water and add them to the digestate in the calibrated glass tube. Then bring the sample to a final volume of 100 ml with DI water and mix well.

11.2.1.1 If calibrated glass tubes are used for digestion and the final volume of 100 ml is brought up in the same tube, be sure to mix well using parafilm.

11.2.1.2 Reagents can be added with a bottle pipettor that is accurate to within 90 to 110%.

11.3 Measure out aliquots of the digested standards and samples into the autosampler cups. Work from the prep log and double check all transfers. Let all samples sit uncovered in the open autosampler vials for a minimum of one minute. Place the racks in the autosampler. Move the stannous chloride line into the stannous chloride bottle.

11.4 Start the calibration.

11.4.1 Click run sequence. The instrument will run the calibration and then pause. Click stop. Go to the Calibration page. Accept the calibration and then print the calibration. Click the Document icon, then choose HG5-PDF. Rename the file as MA*****_cal.

11.5 After the calibration has been accepted, start to run the samples.

11.5.1 For the Hydra AA II, go to the Sequence page. Right click on the first sample (i.e. ICV) and click start from here.

11.5.2 Review the data. Any samples that are over the range of the curve must be diluted with the dilution acid (see 8.1.2) and reanalyzed. It is recommended that any sample analyzed after a sample with a value over the curve be reanalyzed for confirmation. Make sure to bracket every 10 samples with CCV and CCB checks.

11.6 Both paper and electronic reports can be generated using the report option. Never delete any samples from the reports. Electronic reports must be transferred into the LIMS system where the final calculations are done.

11.6.1 Go to analysis-Click result-Click chapter. Then go to report and select report spec. The normal report spec is "ACCUTEST". Click OK. Click on chapter in order to select all samples. Then click report output and then csv.file. Save as MA*****.csv. To print, select printer output and then type the report title (i.e. MA*****) and enter OK.

11.7 The calculations are done in the LIMS as described below. . For soils, the calculation shown below is used. A final volume of 100 ml is used for calculation purposes. (The final volume is factored out since all standards and samples are brought up to the same final volume and standard concentrations are calculated based on 100 ml.)

$$\text{Final sample concentration in mg/kg} = \frac{\text{concentration in the digestate in ug/l x final volume}}{\text{Initial weight in g x (\%solids/100)}}$$

11.8 Review the data in the LIMS, adding comments and accepting results as appropriate.

11.9 Shut down the instruments.

11.9.1 To shut down the Hydra AA II, move the stannous chloride line from the stannous chloride bottle to the 10% HNO3 rinse bottle. Let the system rinse with 10% HNO3 for several minutes. Then switch the line to DI water bottle and let rinse for several more minutes. Let the pump and gas run until the lines are completely dry. Then go to instrument control menu and click off icon for Lamp, Gas and Pump.

12.0 QUALITY CONTROL

Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

12.1 Instrument Detection Limits (IDLs). The instrument detection limits are determined by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of 7 consecutive replicates of a standard solution at a concentration from 3 to 5 times the estimated detection limit. IDLs must be done quarterly (every 3 months) for each instrument.

12.2 Method Detection Limits (MDLs). MDLs must be established using the lab's SOP EQA075.

12.3 Instrument Calibration. The instrument must be calibrated daily or at a minimum of once every 24 hours and each time the instrument is set up. Calibration standards must be digested using the same procedure as the samples. A minimum of a blank and 5 standards are required. The correlation coefficient of the curve must be a minimum of 0.995. The Relative Error percent (RE%) must be calculated for low and mid points of the curve. The Relative Error for the midpoint is 20% and for low point is 30%. These criteria must be documented and attached to each calibration. No samples must be analyzed until all of the calibration criteria are met.

12.3.1 A linear calibration using the equation y = mx + b is applied where m is the slope and b is the intercept. The calibration is not forced through zero.

12.3.2 The correlation coefficient is calculated using the following equation:

$$Correl(X,Y) = \frac{\sum(x-\bar{x})(y-\bar{y})}{\sqrt{\sum(x-\bar{x})^2\sum(y-\bar{y})^2}}$$

where x is the measured absorbance and y is the standard concentration.

12.3.3 If the calibration curve does not meet criteria, and is redigestion, then any samples digested along with that calibration curve must also be redigested.

12.4 Initial Calibration Verification Standard (ICV). During each analysis, a standard from a different source than the calibration standard must be analyzed. This is analyzed at the beginning of the run. For this method, the ICV must be within 10 percent of the true value. When the measurements exceed these control limits, the analysis shall be terminated, and the problem corrected before proceeding. All reported results must be bracketed by compliant QC.

12.5 Continuing Calibration Verification. Analyze the continuing calibration verification solution and the continuing calibration blank at the beginning of each run and after every tenth sample and at the end of the sample run. If the CCV solution is not within 20 percent of the true value, then no samples can be reported in the area bracketed by that CCV. (Note: the exception is if the CCV is biased high and the samples are less than the detection limit. In that case, the samples can be reported with no flag.)

12.6 Continuing Calibration Blank. Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run. If the CCB is not less than the reporting limit, then no samples can be reported in the area bracketed by the failing CCB.

12.7 CRA (Low) Check or LLQC (Lower Limit of Quantitation Check). For all runs, a low check at the level of the reporting limit must be analyzed at the beginning of the run before analyzing any samples, but not before the ICV. A criterion +/- 30% recovery is applied to this low check standard. If this criterion is not met, then all samples associated with this CRA check must be reanalyzed along with a compliant CRA check.

12.7.1 A number of clients have specific program requirements for frequency and recovery ranges on CRA checks. Check with the Metals supervisor for additional information on these programs.

12.7.2 If the CRA is biased high and there is no mercury found in the samples, then the sample results may be reported for mercury. If the CRA is biased high and there is mercury found in the samples, then the samples with Hg at levels ranging from the CCV to the high standard may be reported. Samples with levels of mercury between the CRA and the CCV standard may be biased high and cannot be reported.

12.8 Method Blank. The laboratory must digest and analyze a method blank with each batch of 20 samples. A minimum of one method blank is required for every 20 samples. A sample batch is defined as a maximum of 20 field samples in a preparation batch over a time period of 24 hours. A matrix spike/matrix spike duplicate, matrix spikes and/or duplicate is required every 20 samples. The method blank must contain mercury at less than ½ the reporting limit (< 1/2 RL). If the method blank contains over that limit, the samples must be redigested or reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit. (Note: Not all program codes allow this. Verify prior to proceeding.)

12.9 Lab Control Sample. The laboratory must digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control or spike blank is required for every 20 samples. Note: For soils, if a lab control is not available, a spike blank can be used. For a running batch, a new lab control sample is required for each different digestion day. The laboratory must assess laboratory performance of an aqueous lab control against recovery limits of 80 to 120%. In house lab control limits may also be generated to support these default limits. For solid lab controls, the elements must be within the range of "QC Performance Acceptance Limits" given by the lab control supplier. If the lab control is outside of the control limits for a given element, all samples must be redigested and reanalyzed for that element. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag.

12.10 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. The laboratory must assess the matrix spike recovery against control limits of 80 to 120. (In house control limits are generated annually for information purposes only.). If a matrix spike is out of control, then the sample from which the MS/MSD aliquots were prepared should also be spiked with a post digestion spike. An analyte spike is added to a portion of a prepared sample, or its dilution, and must be recovered to within 80-120% of the known value. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed. Note: Both the matrix spike amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

((Spiked Sample Result - Sample Result)/Amount Spiked) x 100 = matrix spike recovery

12.11 Matrix Spike Duplicate or Matrix Duplicate. The laboratory must digest a matrix spike duplicate or a duplicate sample for a minimum of 1 in 20 samples. Matrix spike duplicates are normally used unless otherwise specified by client requirements. The relative percent difference (rpd) between the matrix spike duplicate and the matrix spike or between the duplicate and the original sample must be assessed. The rpds are calculated as shown below and must be assessed against a limit of 20% RPD. (In house control limits are generated annually for information purposes only.). If a matrix spike duplicate or a duplicate is out of control, then the results must be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the

reporting limit, then the duplicate is considered to be in control. Note: Both the duplicate amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

$$\frac{\text{(Sample Result - Duplicate Result) x 100}}{\text{(Sample Result + Duplicate Result) x 0.5}} = \text{\% RPD}$$

or

$$\frac{\text{(|MS Result - MSD Result|) x 100}}{\text{(MS Result + MSD Result)/2}} = \text{MSD RPD}$$

12.12 Measurement the Relative Error (%RE):
Relative error is calculated using the following equation:

$$\text{\% Relative Error} = \frac{x'i - xi}{xi} \times 100$$

xi = True value for the standard
x'i = Measured concentration of the standard

13.0 DOCUMENTATION REQUIREMENTS

Refer to the laboratory Quality Assurance Manual for additional documentation requirements.

13.1 Sample Worksheets. Digestion data sheets for the Hg soil samples must show all digestion information including the sample ID's, sample weights, bottle numbers, type of heating used, start times, end times, and pressure or temperature, as appropriate for all digestions. All sample information must be clearly entered on these sheets. In addition, any unusual characteristics of the samples or the digestion procedure must be noted in the comments sections. Make sure also that all dilutions are clearly documented.

13.2 Make sure to record thermometer ID, correction factor, and corrected and uncorrected temperatures for all temperature measurements.

13.3 Standards and Reagents. All stocks and reagents must be recorded in the reagent log book. All standards must be recorded on the digestion log with the samples.

13.4 Any run comments must be written on the raw data for the analysis and on the run log in the LIMS.

13.5 Annual bottle calibration verifications must be documented in the Mercury Bottle calibration log.

14.0 DATA REVIEW AND REPORTING

14.1 All samples must be updated to QC batches in the LIMS system. The analyst is responsible for reviewing all data for compliance with the QC outlined in this SOP. They are responsible for making sure that the raw data is fully documented, and it is loaded into the LIMS system. They are responsible for submitting samples for redigestion and reanalysis, when appropriate.

14.2 After the analyst review is completed, the supervisor or a designated reviewer shall review the run for technical compliance to the SOP. The reviewer is also responsible for making sure that the QC calculations are done correctly and that appropriate flags are added.

14.3 After the reviewer completes their review, the data is released for client access in the LIMS. The raw data and the run log are submitted to the area manager. The manager periodically does an additional review on data for technical completeness. Any hardcopy raw data is transferred to the report generation department for scanning and storage. Instrument data is transferred electronically.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 16.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes.

15.2.2 Hazardous aqueous wastes.

15.2.3 Chlorinated organic solvents.

15.2.4 Non-chlorinated organic solvents.

15.2.5 Hazardous solid wastes.

15.2.6 Non-hazardous aqueous wastes.

16.0 ADDITIONAL REFERENCES

16.1 Leeman Hydra AA instrument manual.

16.2 Leeman Hydra AA II instrument manual.

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
12.3	Added criteria for RE%
7.4	Revised to digestion tubes

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed
7.4.1—7.5	Deleted reference to calibrated of Hg bottles.

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
15	8/20/2019	Wei Zhou
16	1/22/20	Wei Zhou
17	2/14/22	Olga Azarian

END OF DOCUMENT

LAB SUPERVISOR: _____

QA OFFICER: Olga V. Gaines

EFFECTIVE DATE: 5-14-2021

TITLE: METALS BY INDUCTIVELYCOUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP) USING SOLIDS STATE ICP
REFERENCE: SW846 6010D, Rev.5, July2018
REVISED SECTIONS: reference,4.0, 6.1, 12.2

1.0 SCOPE AND APPLICATION

1.1 This method is applicable for the determination of metals in water, wipes, sludges, sediments, and soils. Sample matrices are pretreated following SW846 methods for digestion of soil, sediment, sludge, wipe or water samples. Refer to specific digestion SOP's for more information on digestion techniques.

1.2 A variety of metals can be analyzed by ICAP. These include, but are not limited to, Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sr, Tl, Sn, Ti, V, Zn, Li P and Pd W S Bi Zr

2.0 SUMMARY

2.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

2.2 This SOP describes operation of the ICAP 6500 and ICAP 7000 Spectrometer following method SW846 6010D.

2.2.1 This inductively coupled argon plasma optical emission spectrometers (ICP-OES) uses an Echelle optical design and a Charge Injection Device (CID) solid-state detector to provide elemental analysis. Control of the spectrometer is provided by PC based iTEVA software.

2.2.2 In the instrument, samples are nebulized, and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radiofrequency inductively coupled plasma. The spectra are dispersed by a spectrometer, and the intensities of the emission lines are monitored the solid-state detector.

2.2.3 Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. In one mode of analysis the position used must be as free as possible from spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Interferences which cannot be addressed with background correction must be corrected using the appropriate interelement correction factors.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The normal reporting limits for this method have been established at the concentrations listed in Table 1. Reporting limits may vary depending on client needs and lab protocols, but the reporting limits must always be verified with a low check which meets the criteria outlined in this SOP. In addition, the reporting limits must always be greater than the MDL. Refer to the scheduling sheets and check with the metal's supervisor for further information.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method. Refer to SGS SOP EQA075

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed, and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD. The calibration check standard is a mid-range calibration standard.

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards.

SPIKE BLANK SAMPLE. Digest and analyze a laboratory control sample (Soil LC) or spike blank with each set of samples.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX SPIKE DUPLICATE: A matrix spike duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the matrix spike duplicate and the matrix spike must be assessed.

$$\frac{(|\text{Matrix Spike Result} - \text{Matrix Spike Duplicate Result}|) \times 100}{(\text{Matrix Spike Result} + \text{Matrix Spike Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank.

METHOD DETECTION LIMITS (MDLS). The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is distinguishable from method blank results.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.)

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards must be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analysis.

LOW LEVEL CALIBRATION VERIFICATION (CRI or LLCCV). The LLCCV or CRI standard is a check standard containing the elements of interest at (or below) the reporting level for each element.

HIGH STANDARD/LINEAR RANGE: The high standard is a check standard containing elements of the interest at the instrument linear range. The linear range establishes the highest concentration that may be reported without diluting the sample. The acceptance criteria are +/-10 of the true value.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Plan and Personal Protection Policy, which include the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

6.0 PRESERVATION & HOLDING TIME

6.1 All water samples must be preserved with nitric acid to a pH of 2 or less. All solid samples must be stored in a refrigerator at ≤6°C.

6.2 All samples must be analyzed within 6 months of the date of collection.

7.0 INTERFERENCES

7.1 Several types of interferences can cause inaccuracies in trace metals determinations by ICP. These interferences are discussed below.

7.2 Spectral interferences are caused by overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena, and background contribution from stray light from the line emission of high concentration elements. Corrections for these interferences can be made by using interfering element corrections, by choosing an alternate analytical line, and/or by applying background correction points.

7.3 Physical interferences can be caused by changes in sample viscosity or surface tension, by high acid content in a sample, or by high dissolved solids in a sample. These interferences can be reduced by using an internal standard, by making sample dilutions or by analyzing a sample using the method of standard additions.

7.4 Chemical interferences are not pronounced with ICAP due to the high temperature of the plasma, however if they are present, they can be reduced by optimizing the analytical conditions (i.e. power level, torch height, etc.).

8.0 EQUIPMENT AND SUPPLIES

8.1 Currently there are five solid state ICPs available for use in the lab. They are Thermo 6500 and 7000 ICP units. These units have been optimized to obtain low detection limits for a wide range of elements. Since they are solid state systems, different lines may be included for elements to obtain the best analytical results. However, the lines which are normally included in the normal analysis program are shown in Table 2.

8.2 Instrument auto-samplers. For random access during sample analysis.

8.3 Class A volumetric glassware and pipets.

8.3.1 All glassware must be washed with soap and tap water and then soaked in a 10% nitric acid bath for a minimum of 2 hours. It must then be rinsed at least 3 times with deionized water.

8.4 Glass autosampler tubes

8.4.1 Autosampler tubes must be washed with soap and tap water and then soaked in a 10% nitric acid bath for a minimum of 2 hours. They must then be rinsed at least 3 times with deionized water.

8.5 Autopipettes with tips. These must be calibrated and checked as outlined in the autopipette SOP, EQA004.
8.6 ASXPRESS PLUS Valve system from CETAC
8.7 Argon humidifier

9.0 REAGENTS

9.1 All chemicals listed below are reagent grade unless otherwise specified. Deionized water must be used whenever water is required. The expiration date for standards and reagents is the date supplied by the manufacturer or if no expiration date is given, a default of 6 months is used. For acid solutions (nitric, sulfuric, hydrochloric) the expiration date is 2 years from the date of preparation of the solution.

9.2 Hydrochloric acid, trace metals grade.

9.3 Nitric Acid, Baker instra-analyzed or equivalent.

9.4 Standard stock solutions available from Absolute, Inorganic Ventures, CPI, Ultra Scientific or equivalent.
Note: All standards must be ICP quality standards.
Calibration Standards: These are made up by diluting the stock solutions to the appropriate concentrations. Fresh calibration standards must be prepared every day.

9.4.1 Standards must be approximately matrix matched to the samples. For most samples, a 5 percent nitric acid and 5 percent hydrochloric acid will approximate the acid matrix of the sample and limit nebulization problems. If it is known that the samples contain a significantly different acid matrix, then the matrix of the standards must be modified, or the samples must be diluted so that they are in a similar matrix to the curve.

9.4.2 Standards must be prepared so that there is minimal spectral interference between analytes. See Table 10 for the make-up and concentrations of standards and stock solutions being used to calibrate the ICP. The standard curve consists of a blank and 1 non-zero standard at the levels shown in Table 10.

9.5 Calibration/Rinse Blank. The calibration blank is prepared by diluting a mixture of 50 ml of concentrated nitric acid and 50 ml of concentrated hydrochloric acid to a final volume of 1 liter with deionized water.

9.6 Analytical Quality Control Solutions. All the solutions below are prepared by adding either mixed or single element metals solutions to a solution containing 5 percent nitric acid and 5 percent hydrochloric acid and diluting to a fixed final volume with this acid mixture. These solutions must be placed in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles for long term storage.

9.7.1 Initial Calibration Verification solution (ICV). This standard solution must be made from a different source than the calibration curve. The values for each element must be near the midpoint of the calibration curve. This solution is used to verify the accuracy of the initial calibration. See Table 4 for suggested ICV concentrations.

9.7.2 Continuing Calibration Verification solution (CCV): The metals concentrations for this standard must be at approximately the mid-point of the calibration curve for each element. This standard must be prepared from the same source that is used for the calibration curve. See Table 4 for suggested CCV concentrations.

9.7.3 Interference Element Check Solutions. These solutions must be used on a periodic basis to check the interfering element corrections on the instruments and interfering element solutions may be modified. Two acceptable solutions are outlined below.

9.6.1.1 ICSA Solution (Interference Check Standard) This is a QC standard used to verify the accuracy of the interferents (Al, Ca, Fe, and Mg) and the accuracy of the inter-element correction factors applied in the absence of analyte. The recommended concentrations are shown below. If the linear ranges on a given instrument are lower than these levels, the concentrations may be set near the top of the linear range for those elements.

Al	500 mg/L
Ca	400 mg/L
Fe	200 mg/L
Mg	500 mg/L

9.6.1.2 ICSAB Solution (Interference Check Standard with analytes): The ICSAB solution contains both the interferents and the analytes of interest. The recommended concentrations are shown below. If the linear ranges on a given instrument are lower than these levels, the concentrations may be set near the top of the linear range for those elements

Ag	1.0 mg/L	Zn	1.0 mg/L
Ba	0.50 mg/L	As	1.0 mg/L
Be	0.50 mg/L	Se	1.0 mg/L
Cd	1.0 mg/L	Sb	1.0 mg/L
Co	0.50 mg/L	Tl	1.0 mg/L
Cr	0.50 mg/L	Mo	0.5 mg/L
Cu	0.50 mg/L	P	0.5 mg/L
Mn	0.50 mg/L	Al	500 mg/L
Ni	1.0 mg/L	Ca	400 mg/L
Pb	1.0 mg/L	Fe	200 mg/L
V	0.50 mg/L	Mg	500 mg/L
W	0.50 mg/L	Zr	0.50 mg/L

Li	0.50 mg/l	Sr	0.5 mg/l
Bi	0.50 mg/l	Ti	0.5 mg/l
B	0.50 mg/l	S	0.5 mg/l
Sn	0.50 mg/l	Si	0.5 mg/l

9.6.2 CRI Standards (also referred to as LLCCV). The CRI standard must contain the elements of interest at (or below) the reporting limit for each element. The CRI level is at the reporting limit as shown in Table 1. This solution is to be prepared at the reporting limit level for each element. They must be made in the same matrix as the calibration standards. Note: The CRI must be verified at the RL before any dilutions are applied

9.7 Matrix Spike and Spike Blank Solution: The final concentrations suggested for the matrix spike and spike blank solutions are shown in Table 5. Refer to METALS SPIKING SOLUTION AND STANDARDS PREPARATION SOP EMP 202 Table 1,1A, and 3 for preparation/concentration and amount of spiking solutions. Appropriate amount of the resulting stock solution is added to the matrix spike and blank spike samples before they are digested.

9.8 Matrix Spike and Spike blank (For aqueous samples and TCLP leachates).

9.8.1 The final concentrations suggested for the matrix spike are shown in Table 5. Spiking solutions, they are prepared by adding either mixed or single element metals solution. Refer to METALS SPIKING SOLUTION AND STANDARDS PREPARATION SOP EMP 202 Table 1,1A, and 3 for preparation/concentration and amount of spiking solutions. Resulting stock solution is added to the matrix spike and blank spike samples before they are digested.

9.8.2 The Spike blank sample must be digested and analyzed for every batch of 20 samples or less. The Blank spike prepared by adding either mixed or single element metals solutions to DI water and bringing up to a fixed final volume. For TCLP samples, the blank spike must be made using blank leachate solution rather than DI water. 50 ml of this solution is digested and brought to a final volume of 50 ml.

9.9 Liquid Argon or Argon Gas. Argon is provided by Air Products in the large outdoor tank. No lab monitoring of the tank is normally necessary

9.10 Internal Standard Solution (with matrix modifier). To a 2-liter flask containing approximately 1500 ml of DI water, add 40 ml of 10,000 mg/l Cesium solution, 10 ml of 10000 mg/l indium, and 2 ml of 10000 mg/l yttrium. Add 100 ml concentrated nitric acid and 100 ml concentrated hydrochloric acid and bring to a final volume of 2000 ml and mix well. This solution is added to all samples and standards as the instrument is running using a split line on the peristaltic pump.

10.0 PROCEDURE

10.1 General procedure on how to operate the SS Trace is described below. Refer to the Thermo 6500-7000 operation manual for further details.

10.2 Before bringing up the instrument, make sure that the lines, the torch, the nebulizer, and the spray chamber are clean, the dehumidifier is filled with DI water up to the level between Minimum and Maximum, and that there are no leaks in the torch area.

10.3 Turn on the recirculating cooler. Verify that the liquid argon is turned on.

10.4 Set up the pump tubing and engage the peristaltic pump.

10.5 Put a new solution of acid rinse into the rinse reservoir. (Note: the composition of the rinse solution may be periodically changed to minimize sample introduction problems and sample carryover.) If internal standard is being used, make sure that sufficient internal standard solution is prepared.

10.6 Start up the instrument following the sequence shown below.

10.6.1 Double click the **iTEVA Control Center** Icon on desktop. Type ***admin*** in User Name field, and then click OK.

10.6.2 Once the iTEVA Control Center window is opened, click on **Plasma** Icon at status bar area. Then click on **Instrument Status** to check the interlock indicators (torch compartment, purge gas supply, plasma gas supply, water flow and exhaust must be in green; drain flow and busy must be in gray) and the Optics Temperature. (It needs to be around 38°C.) Click on the Close box.

10.6.3 Click **Plasma On**. After the plasma is on, close the Status window and let the instrument warm to up for 15 to 20 minutes before starting the analysis. New tubing may take an hour to stabilize.

10.7 Torch Alignment and Auto Peak

10.7.1 If the torch has been cleaned, then it must be realigned after it is replaced

10.7.1.1 Open the method and then click on **Sequence** tab, and then click on List View Icon until you reach rack display.

10.7.1.2 Send probe to the cup which is filled with 2 ppm Zn solution in Autosampler program. eg. Go to S-6 position (you can assign any position in the rack for torch alignment) which is filled with 2 ppm Zn solution.

10.7.1.3 Click on **Analysis** tab and then select **Torch Alignment** from **Instrument** drop down menu. There will be a popup dialog box present. Click RUN. Then there will be another dialog box pop up (This is a reminder for Torch Alignment Solution (2 ppm Zn)), let the solution to pass through the plasma and click Ok. Now, the instrument is initiating an automated torch alignment. It takes about 7 minutes to complete this step. Progress is indicated in the progress bar.

10.7.1.4 After Torch Alignment is done, click Close. Click on **Sequence** tab and then follow by **List View** Icon.

10.7.1.5 Go to Rinse position at rack display, right click to select Go to rinse and let it rinse for 2 minutes.

10.7.2 Perform Auto Peak.

10.7.2.1 It is recommended that the Auto Peak Adjust procedure be performed monthly or whenever the peak shape has shifted for any element. A standard that contains all of the lines of interest is used and the system automatically makes the appropriate fine adjustment. (CCV solution is used for this process.)

10.7.2.1.1 A shift in peak shape can be defined as when the peak is no longer in the middle of the defined viewing window. The window must be set so that the peak is approximately centered and there is a sufficient area measured so that reproducible, consistent data can be obtained at reporting limit levels. This is done as part of the automatic process, but the window size can be adjusted manually in the method based on the shape of the peak to obtain the best fit

for the peak. A wider peak may need a broader integration window for the best analysis. In general, the window must cover at least the top 1/3 of the peak.

10.7.2.2 Click **Sequence** tab and then click on **List View** Icon till the rack is displayed.

10.7.2.3 Send the probe to the cup which is filled with CCV solutions using Autosampler program (you can assign any position in the rack which has CCV solution filled for auto peak adjust) then click on **Analysis** tab. All elements' result is showed in the display area. From **Instrument** drop down menu, select **Perform Auto Peak**. There will be a popup dialog box present. Highlight _All Elements, then click RUN. Then there will be another dialog box pop up (This is a reminder for Perform Auto Peak Solution), click Ok. Now, the instrument is performing auto peak adjust. It takes about 5 minutes to complete this process. The Auto Peak dialog box will show a green " √" in front of All Elements, which indicates Auto Peak is completed.

10.8 Open the method and start up the run.

10.8.1 Click on **Analyst** Icon at the workspace. Go the Method and choose Open from the drop-down menu. Select the method with a Revision (usually select the last revision used).

10.8.2 Go to **Method** tab at the bottom of left-hand corner to click on **Automated Output** at the workspace area. Type a filename in Filename field in the data display area (i.e.: SA073107M1: starts with SA, then follow by MM-DD, then M1; M1 indicates the first analytical run for that day, then follow by M2, M3 and so on for the second and third runs).

10.8.3 Click on **Sequence** tab at the bottom of left-hand corner. From **Auto-Session** drop down menu bar, click on **New Autosampler** to create a sequence. This will pop up a dialog box, then click on **New** and fill number of samples (i.e.: 100) in the Number of Samples field and the sample ID (usually leave this field empty) in Sample Name field. Type a sequence name (i.e.: SEQ073107M1: starts with SEQ, then MM-DD-YY, then M1; M1 indicates the first analytical run for that day, then follow by M2, M3 and so on for the second and third runs) in the Sequence Name field. Click OK, then put in "0" on Settle Time between the Sequences box, click OK.

10.8.4 Right click on **Untitled** (CETAC ASX-520 Enviro 5 Named Rack is the rack that currently using) at the workspace area, click on **Auto-Locate ALL** to locate all samples.

10.8.5 Double click on **Untitled** again, then click on the sequence name (i.e.: SEQ073107M1), on the data display area, type the sequence in Sample name column, dilution factor (if needed) in CorrFact column, check the box in front of Check column, and select an appropriate check table.

10.8.6 Once done with creating sequence, go to **Method** drop down menu and save all changes as **Save As**. There will be a Save a Method dialog box present, go to Save Option to check on "Overwrite Method and bump revision number" box, then click OK.

10.8.7 Go to **Sequence** tab, click on **List View** Icon from tool bar, then click on **Connect Autosampler to PC and Initialize** Icon. (Now, the autosampler tip is up and sits on the top of the rinse cup.)

10.8.8 The sequence includes the calibration and run quality control.

10.8.8.1 Calibrate the instrument as outlined below using the standards shown in Table 3. This calibration procedure is done a minimum of once every 24 hours. The calibration standards may be included in the autosampler program or they may be run separately.

10.8.8.2 Analyze ICV and ICB after the calibration is completed and before any samples are analyzed.

10.8.8.3 For mixed runs (EPA 200.7 and SW846 6010D), the first CCV is designated the ICCV. For samples and quality control, insert the list pointer after a space after the sample. Check with the metal's supervisors for additional information on the use of list pointers. In general, list pointer 7 refers to the SW846 6010D method and list pointer 1 refers to EPA 200.7 method.

10.8.8.4 Low Level Calibration Verification (Low checks or LLCCV) Run low checks at reporting limit levels after ICCV and CCB. The low checks are named as CRI (or CRIB for DOD run), CRID and CRIA. The levels for each low check are listed in Table 6, Table 7 and Table 8.

10.8.8.4.1 Muti-level low check solutions must be analyzed for default reporting limits and special client reporting limits.

10.8.8.4.2 Method limits of 80 to 120% are applied to the CRI low check standard.

10.8.8.5 Before analyzing any real samples, an interference check solution (ICSA-ICSAB) must be checked. For all spiked elements, the analyzed results must be within 20 percent of the true results. For unspiked elements, the interfering element solutions must contain **less than** the absolute value of the reporting limit for each element.

10.8.8.6 If the interfering element solution is not within specifications and that element must be reported, then new interfering element correction (IEC) factors will need to be generated following the procedure outlined in Section 11 below. If new IEC's are generated, then the run must be restarted from the ICSA, ICSAB quality control samples and new CCV checks must be run before any samples can be reported.

10.8.8.7 After the initial analytical quality control has been analyzed, the samples and the preparation batch quality control must be analyzed. Each sample including calibration standards and QC needs at least 3 replicates using at least 5 seconds integration time, this time can be modified. For samples containing levels of elements greater than approximately 5 times the reporting limits, the relative standard deviations for the replicates must be less than 5%. If not, reanalyze the sample. Upon reanalysis, the RSDs are acceptable then report the data from the reanalysis. If RSD's are not acceptable on reanalysis, then the results for that element must be evaluated by the data reviewer and footnoted if necessary. In some cases, an additional dilution analysis may be needed. Check with the area supervisor or manager for additional information.

10.8.8.8 Between each sample, flush the nebulizer and solution uptake system with a blank rinse solution for 60-120 seconds to ensure that analyte memory effects are not occurring. A time of 60-120 seconds is recommended for most analyses. When using Sprint valve and ASX EXPRESS Plus unit the uptake and rinse timing are being controlled by the valve configurator, Follow the manufacturer guideline. Normal timing for the valve configurator is as follow.

Loop Evacuation Delay: 1-3 Sec
Loop Load: 5-11 Sec
Time to Evacuate: 1-3 Sec
Rinse Station Fill: 5-10 Sec
Rinse Evacuation Delay: 1-3 sec
Equalization Delay: 1-3 Sec
Probe Rinse: 5-10 Sec

10.8.8.9 Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth samples during an analysis run, whichever is more frequent, and at the end of the sample run.

10.8.8.10 If the CCV solution is not within 10 percent of the true value, no samples can be reported in the area bracketed by the failing CCV for the failing element. Additionally, for the elements with a CCV greater than 5 times the reporting limit, the relative standard deviation for the replicates must be less than 5 percent.

10.8.8.11 The CCB results must be less than the reporting limit or limit of quantitation for each desired target analyte. If these criteria are not met, then no samples can be reported in the area bracketed by the failing CCB for the failing element and all samples must be submitted for reanalysis.

10.8.8.11.1 However, if the samples are high relative to the CCB (> 10 X the CCB level) and a higher reporting limit is acceptable for the final end use of the data, then the samples may be evaluated using a higher reporting limit to meet the CCB criteria. This must be clearly documented on the run if a higher reporting limit is applied.

10.8.8.11.2 In addition, at the reviewer's discretion, samples that are < RL may be reported when the CCB is biased high. Analysts must assume that samples bracketed by a failing CCB must be reanalyzed unless instructed otherwise.

10.8.8.11.3 If a CCB fails, if possible, the analyst must stop the run and run a new CCV, CCB pair before proceeding with the analysis of any additional samples.

10.8.8.12 For one sample per preparation batch a serial dilution must be prepared. Normally the sample used for the serial dilution is the sample that is used for the matrix spike and matrix spike duplicate. For the serial dilution, a 1:5 dilution must be made on the sample.

10.8.8.13 When matrix spike or matrix spike duplicate is out of acceptable limits, then post-digest spikes be prepared to determine potential interferences.

10.8.8.14 For any readings that exceed the linear range for a given element, a dilution is required. After a high reading, the sample following the high one must be examined for possible carryover. The verifications may be necessary by rinsing the lines with an acid solution and then rereading the sample. A limit check table may be built into the autosampler file so that samples exceeding the linear range are flagged on the raw data.

10.8.8.15 For the interelement spectral interference corrections to remain valid during sample analysis, the interferent concentration must not exceed its linear range. If the interferent exceeds its linear range or its correction factor is big enough to affect the element of interest even at a lower concentration, sample dilution with reagent blank and reanalysis is required. In these circumstance analyte detection limits are raised.

10.8.8.16 Anytime that the interference is large relative to the sample, dilution may be required.

10.8.8.17 For any readings where the internal standard is outside of the range of 70 to 130% of the internal standard level in the calibration blank, then the sample must be diluted until the internal standard is within that range. See Table 11 for the assigned Internal Standard for each element.

10.8.9 This method does not require the analysis of an interfering element check solution at the end of the run. However, this may be required to meet other method and/or client requirements. Run the ICSA and ICSAB solutions as instructed by the Metals lab supervisor or manager or as noted in the program code instructions.

10.8.10 After the instrument is optimized, click **Run Auto-Session** Icon to start the run.

10.8.11 If you need to add or delete samples once the run is started, follow the steps shown below.

10.8.11.1 Adding Samples.

10.8.11.1.1 Click on **Sequence** tab, then click on **List View** Icon at the tool bar. There is the sequence table on the data display area.

10.8.11.1.2 Click on **Add Samples** Icon. This will pop up the dialog box, then fill number of samples that need to add in field. Click OK. By doing this, samples will be added at the end of sequence without a location the rack.

10.8.11.1.3 Go to the added samples, on the To position ID column, assign a number for each sample. This number will be the position in the rack. On the Samplename column, type in sample IDs, fill in Corr Fact (if needed) and Check Table.

10.8.11.1.4 The added samples will be analyzed at the end of the original sequence run order unless you assign them to run under different order.

10.8.11.2 Deleting Samples.

10.8.11.2.1 Click on **Sequence** tab, then click on **List View** Icon under the sequence display area.

10.8.11.2.2 To the sample that need to be deleted, on the to position ID column, change the number to "0". By doing this, that sample will be unlocated in the rack and the autosampler tip will go to the next sample.

10.9 When the analysis is completed export the data to LIMS following the procedure outlined below.

10.9.1 Double click on **ePrint** Icon on desktop. There will be a LEADTOOLS ePRINT dialog box pop up, then click **Finish Jobs** and **OK** boxes.

10.9.2 Double click the **PDF** Icon on desktop, the PDF file will present as Document_#. Right click on that file, select **Rename** to change the file name to an assigned analytical run ID. (i.e.: MA8324). This is the raw data for MA8324.

10.9.3 Drop the raw data to LIMS.

10.9.4 By completing above steps, the raw data (i.e.: MA8324) can be pulled up in the Raw Data Search function.

10.9.5 For any Thallium hit found during the data review or analysis, the sample will be rerun to confirm. If the hit is confirmed, check the thallium spectrum of that sample to determine if the hit is caused by the matrix interference or true. If the spectrum indicates any matrix interference, then the sample will be rerun on dilutions to bring the thallium result to <RL

10.10 The data must be reviewed in the LIMS as outlined in the inorganic data review SOP, EQA034. Calculations for water samples are done automatically in the LIMS using the equation shown below.

original sample concentration of metal (μg/l) =

$$\frac{\text{(conc. in the digestate (μg/l)) x (final digestate volume (ml))}}{\text{(Initial sample volume (ml))}}$$

10.11 Aft the end of the analysis day, the ICP must be brought down using the following sequence:

10.11.1 Place the autosampler tip in rinse cup and rinse in a mixed solution of 5% nitric acid and 5% hydrochloric acid for 10 minutes and in DI water for 20 minutes. **Note**: A stronger acid may be needed depending on the matrix of the samples that were analyzed.

10.11.2 Turn off the plasma by click on the **Plasma** Icon and click on **Plasma Off**.

10.11.3 Close all iTEVA programs/ windows.

10.11.4 Release the tension on the sample pump platen.

10.11.5 Switch off recirculating chiller.

11.0 PROCEDURE FOR GENERATION OF INTERFERING ELEMENT CORRECTION FACTORS

11.1 All IEC's must be verified and updated a minimum of once every 6 months or whenever instrument conditions change significantly. It is recommended that elements with frequent high concentrations or with large IEC's must be checked more frequently.

11.2 Calculate the IEC correction factors and enter them into the method. Verify that the recalculated sample results are within QC limits. Calculate the correction factor using the equation shown below. This correction factor must be added to the correction factor already in place in the method for a given element.

$$\text{IEC} = \frac{\text{Concentration Result of the element with the interference}}{\text{Concentration result of the interfering element}}$$

11.3 Analyze the ICSA/ICSAB solutions and/or SIE solutions and verify that the combined standards are within QC limits. If they are not, make additional changes to the IEC factors and then re-verify both the individual and combined solution values.

11.4 Save and update the method.

11.5 Interfering element correction factors saved as raw data along with the run printouts daily so that the IEC's for a given run are traceable.

12.0 QC REQUIREMENTS

12.1 This section outlines the minimum QA/QC operations necessary to satisfy the analytical requirements for method SW846 6010D.

12.2 Method Detection Limits (MDLs). Initial MDL must be verified quarterly and re-calculated once every thirteen months. Refer to SGS SOP EQA075.

12.3 Instrument Detection Limits (IDLs). Instrument Detection Limits (IDLs). It is required that IDL's be completed **ANNUALLY** or after major instrument maintenance or by project specifications. The Instrument Detection Limits (in ug/L) are determined by analyzing 10 replicates of a reagent blank solution. The IDL is defined as 3 times the standard deviation of 10 reps added to the mean of the replicates; as long as it is 0 or positive (Use zero for the mean if the mean is negative.). IDLs shall be determined and reported for each wavelength used in the analysis of the samples.

12.4 Linear Calibration range: The linear range establishes the highest concentration that may be reported without diluting the sample. Following calibration, analyze a standard at a higher concentration than the high standard in the calibration. The standard must recover within 10% of the true value, and if successful, establishes the linear range. The linear range standards must be analyzed in the same

instrument run as the calibration they are associated with (i.e., on a daily basis) but may be analyzed anywhere within that run. If a linear range standard is not analyzed for any specific element.

12.5 Initial Calibration Verification (ICV) and Initial Calibration Blank (ICB). After every new calibration, an ICV must be analyzed. The analysis of the ICV may be followed by the analysis of the ICB,

12.5.1 For the ICV, all elements to be reported must be within 10 percent of the true value and the replicates that exceed 5 times the reporting limit must have a relative standard deviation of less than 5 percent. The ICV must be from a different source than the calibration standards and must be near the mid-point of the calibration curve. If the ICV does not meet criteria, then the problem must be identified and corrected before samples can be run and reported for the element(s) that are outside of criteria. Correction of the problem can be verified by rerunning the check standard and showing that it meets QC criteria.

12.5.2 If an ICB is analyzed, then all elements to be reported must be less than ½ of the RL (LLOQ). If the ICB is outside of criteria, then the problem must be identified and corrected before samples can be run and reported for the element(s) that are outside of criteria. Correction of the problem can be verified by rerunning the check standard and showing that it meets QC criteria.

12.6 Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB). Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run.

12.6.1 For the CCV, all elements to be reported must be within 10 percent of the true value and the replicates that are greater than 5 times the reporting limit must have a relative standard deviation of less than 5 percent. The CCV must be made from the same source as the calibration standards at a concentration near the mid-level of the calibration curve. If an element does not meet the recovery criteria of the CCV (90 to 110%), then no samples can be reported for that element in the area bracketed by the CCV. Relative Error (%RE) must be 10% for CCV (See section 12.20 for calculation)

12.6.1.1 If the replicate RSD is high, but all replicates are within the recovery limits, then the results can be accepted at the discretion of the reviewer.

12.6.2 For the CCB, all elements to be reported must be less than the reporting limit (LLOQ). If an element does not meet this criterion then no samples can be reported for that element in the area bracketed by the CCB.

12.7 Interference Check Standard (ICSA-ICSAB). An interference check standard must be analyzed at the beginning of each analytical run. For all spiked elements, the analyzed results must be within 20 percent of the true values. For unspiked elements, the interfering element solutions must contain less than the absolute value of the reporting limit for each element. If these criteria are not met, then no samples containing the elements in question can be reported in the area bracketed by this QC unless the samples contain no significant interferents.

12.8 Low Level Calibration Verification (CRI, CRIB, CRID, CRIA or LLCCV). These are the low-level calibration verification standards containing the elements of interest at (or below) the reporting level for each element. A low-level check standard at or below the RL/LOQ must be analyzed at the beginning of each calibration (analysis) batch. The acceptance criterion for these checks is 80 to 120% recovery. Relative Error (%RE) must be 20% for LLCCV (See section 12.20 for calculation).

12.8.1 The low-level calibration verification is initially verified by the analysis of at least 7 replicate samples, spiked at the RL/LOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement on precision and accuracy at the LLOQ. In most cases the mean

recovery must be +/- 35% of the true value and RSD must be < 20%. Ongoing quarterly verification is required.

12.8.2 More frequent LLCCV checks may be analyzed during the course of the run if system stability at the low end of the calibration is questionable or if the lab wants to ensure that fewer samples will have to be submitted for reanalysis if there is a failed low check at the end of a run.

12.9 Method Blank: The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20-sample batch. All elements to be reported must be less than ½ of the RL (LLOQ). if the samples are high relative to the Method blank (> 10 X of the level), then the samples may be reported. If the method blank does not contain target analytes at a level that interferes with the project specific DQOs, then the method blank is considered acceptable.

12.9.1 The default 6010D method limit for the method blank is that is must be less than one half of the reporting limit.

12.9.2 If the method blank does not meet criteria, then it can be reanalyzed along with any associated samples. If it is still unacceptable, then all associated samples must be redigested and reanalyzed along with the other appropriate batch QC samples

12.10 Spike Blank: The laboratory must digest and analyze a spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20-sample batch. The laboratory must assess laboratory performance of the spike blank against recovery limits of 80 to 120 percent. In house spike blank limits may also be generated to support these default limits. If the spike blank is outside of the control limits for a given element, all samples must be redigested and reanalyzed for that element.

12.10.1 If solid lab controls are used, then the manufacturer's QC Performance Acceptance Limits must be applied.

12.11 Matrix Spike: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Recoveries must be assessed against default limits of 75 to 125 percent. In house limits may be generated for this method for informational purposes only Note: Both the matrix spike amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

((Spiked Sample Result - Sample Result) / Amount Spiked) x 100 = matrix spike recovery

12.11.1 Post-digest spike must be performed when matrix spike is out of the limits of 75 to 125 percent.

12.12 Matrix Spike Duplicate (MSD) or Matrix Duplicate DUP). The laboratory must digest a matrix spike duplicate or matrix duplicate sample for a minimum of 1 in 20 samples. The relative percent difference (RPD) between the MSD and the MS or between the DUP and the sample must be assessed. The RPD is calculated as shown below. The control limit for the duplicate RPD is method defined as 20%. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of $\pm$ the reporting limit, then the duplicate is considered to be in control. Note: Both the duplicate amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

12.12.1 If an MSD or duplicate is out of control, then the data must be checked carefully to confirm that the high rpd for a given element is not a result of an analytical problem. If an analytical problem is suspected, the MSD or duplicate must be reanalyzed for confirmation. If the initial and reanalysis are in agreement (within 20%), then the high rpd is a result of preparation or sample issues and further analysis of the initial preparation is not required. If

the initial and reanalysis are not in agreement due to an analytical problem, then any affected samples in the associated batch must also be reanalyzed for that element.

12.12.2 If more than 50% of the elements in a sample (that have levels of at least 5 times the reporting limit) have a high RPD, then the MSD or duplicate must be redigested for confirmation, unless the sample matrix is such that the non-homogeneity of the sample is visually apparent. If the results confirm, the results from the original MSD or duplicate must be flagged as indicative of possible sample non-homogeneity. If the results do not confirm, then the whole batch must be digested and reanalyzed.

12.12.3 If 50% or less of the elements in a sample (that have levels of at least 5 times the reporting limit) have a high rpd, then the high rpd(s) must be footnoted as indicating possible sample non-homogeneity unless other problems are suspected. If problems are suspected, the reviewer will initiate redigestion and reanalysis of the batch.

12.12.4 The calculations used to calculate RPD are shown below.

$$\frac{(|\text{MS Result} - \text{MSD Result}|) \times 100}{(\text{MS Result} + \text{MSD Result})/2} = \text{MSD RPD}$$

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

12.13 Serial Dilution. A serial dilution is required on a frequency of one in 20 samples. For one sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution must be prepared. Normally the sample used for the serial dilution is the sample that is used for the matrix spike and matrix spike duplicate. For the serial dilution, a 1:5 dilution must be made on the sample. The results of the 1:5 dilution must agree within 20 percent of the true value as long as the sample is greater than 25 times the reporting limit for that element before dilution and the sample results are within the linear range. If not, an interference effect must be suspected, and the serial dilution result for the element with the suspected interference must be footnoted. The serial dilution is calculated as shown below.

$$\frac{100 \times ((\text{Sample result} - \text{Serial dilution result}))}{\text{Sample result}} = \text{Serial dilution percent difference}$$

12.14 Post Digestion Spike: Post-digest spikes used to determine potential interferences. The test only needs to be performed for the specific elements that failed original matrix spike limits, and only if the spike concentration added was greater than the concentration determined in the unspiked sample.
Using unspiked QC sample, spike with a known quantity of target elements. Post spike concentration is at matrix spike concentration. See TABLE 5 for suggested concentrations of Metals in the Post digestion spike and Table 18 for preparation of post spike. The recovery of the post-digestion Spike must fall within 75 to 125 % acceptance range, relative to the known true value. If the post-digestion MS recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values.
((Post spiked Sample Result - Sample Result) / Amount Spiked) x 100 = Post spike recovery

12.15 For TCLP samples, a post-digest spike for any element where the matrix spike recovery is less than 50% and the sample concentration for that element is within 20% of the appropriate regulatory level and not over the regulatory level. If the post-digest spike passes within limits of 75 to 125% recovery, the low recovery will be attributed to a sample matrix effect at digestion and no further analyses will be required. If the post-digest spike fails, then a second post-digest spike will be prepared on a sample dilution. If this spike passes, all samples will be rerun at this dilution level to confirm the sample results. If the post-digest spike fails again on the diluted sample, then Method of Standard Additions (MSA) will be performed for this sample.

12.16 IEC Correction Factor Generation. All interfering element correction factors (IEC's), must be verified and updated a minimum of once every 6 months or whenever instrument conditions change significantly. The result of <2 times RL (LLOQ) is required on IEC.

12.17 Lower Limit of Quantitation check sample (LLQC). The LLQC is a sample at the reporting limit that is taken through the entire preparation and analytical process. This standard must be analyzed when reporting limits are initial established and on an as needed basis after that. The LLQC is equivalent to the LOQ (Limit of quantitation) standard which must be analyzed quarterly for the DOD QSM program. The limits of quantitation are verified when all analytes in the LLQC sample are detected within 20% of their true value. If the limits cannot be verified at the spiked level, then the quantitation limit must be adjusted to a level where verification is successful.

12.18 Calibration Curve. The calibration curve must be prepared daily using a minimum of a calibration blank and one non-zero. The calibration must be verified with LLCCV/CRI and an ICV before any samples can be analyzed. If the curve is not verified as described in section 12.5 or 12.8, then no results can be reported for those elements which did not meet quality control criteria.

12.19 HIGH STANDARD: The high standard is required in run and acceptance criteria are +/-10 of the true value. See HSTD table 14 for preparation and true value.

12.20 Measurement the Relative Error (%RE):

Relative error is calculated using the following equation:

$$\% \text{ Relative Error} = \frac{x'i - xi}{xi} \times 100$$

xi = True value for the standard
x'i = Measured concentration of the standard
The Relative Error for the CCV is 10% and for LLCCV (CRI) is 20%

13.0 CALCULATIONS

13.1 For water samples, the following calculations must be used. Refer to the QC section for the calculations to be used for the QC samples.

original sample concentration of metal (μg/l) =

$$\frac{\text{(conc. in the digestate (μg/l)) x (final digestate volume (ml))}}{\text{(Initial sample volume (ml))}}$$

13.2 For soil samples, the following calculations must be used.

concentration of the metal in the dry sample (mg/kg) =

$$\frac{\text{(conc. in the digestate (mg/l) x final digestate volume(L))}}{\text{(sample wt. (kg)) x (\% solids/100)}}$$

14.0 DOCUMENTATION REQUIREMENTS

14.1 If any samples or QC checks require reanalysis, a brief explanation of the reason must be documented in the raw data. All instrument data must be exported to the LIMS system and a copy of the run log must be included in the logbook by the instrument.

14.2 The Standard Preparation Logbook must be completed for all standard preparations. All information requested must be completed. The SGS Lot Number must be cross-referenced on the standard vial.

14.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. A copy of any outside maintenance reports must also be kept in the log. In addition to the maintenance, the maintenance log must also contain daily information on such items as the profile intensity. Each instrument has a separate log.

14.4 Any corrections to laboratory data must be done using a single line through the error and a reason for the correction. The initials of the person and date of correction must appear next to the correction.

14.5 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information must also be verified during this review.

15.0 INSTRUMENT MAINTENANCE

15.1 Recommended periodic maintenance includes the items outlined below.

15.1.1 Change the pump tubing weekly or as needed.

15.1.2 Clean the filter on the recirculating pump approximately once a month and dust off the power supply vents every one to two weeks.

15.1.3 Clean the radial view quartz surface weekly or more often if needed.

15.1.4 Clean the nebulizer, torch, and injector tube every two to four weeks or more often as needed.

15.1.5 Change the sampler tip as needed (every one to two months).

15.1.6 Clean the recirculating pump lines every 3 months or more often if needed.

15.1.7 Clean the slides on the autosampler with methanol and wipe them with a Kim Wipe saturated with Teflon spray a minimum of once per day.

16.0 POLLUTION PREVENTION & WASTE MANAGEMENT

16.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 16.2.

16.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

16.2.1 Nonhazardous aqueous wastes.
16.2.2 Hazardous aqueous wastes
16.2.3 Chlorinated organic solvents
16.2.4 Non-chlorinated organic solvents
16.2.5 Hazardous solid wastes
16.2.6 Non-hazardous solid wastes

17.0 ADDITIONAL REFERENCES

17.1 Refer to other SOP's for ICP analysis (CLP, and EPA 200.7 for both DW and WW).

TABLE 1: NORMAL REPORTING LIMITS BY ELEMENT				
ANALYTE	WATER & WIPE REPORTING LIMIT(µg/l)	SOIL REPORTING LIMIT (mg/kg)	SOIL REPORTING LIMIT (ug/l)	TCLP REPORTING LIMIT
Aluminum	200	50	500	
Antimony	6	2	20	
Arsenic	3	2	20	0.50
Barium	200	20	200	1.0
Beryllium	1	0.2	2	
Cadmium	3	0.5	5	0.025
Calcium	5000	500	5000	
Chromium	10	1	10	0.05
Cobalt	50	5	50	
Copper	10	2.5	25	
Iron	100	50	500	
Lead	3	2	20	0.50
Magnesium	5000	500	5000	
Manganese	15	1.5	15	
Nickel	10	4.0	40	
Potassium	10000	1000	10000	
Selenium	10	2	20	0.50
Silver	10	0.5	5	0.05
Sodium	10000	1000	10000	
Thallium	10	1	10	
Vanadium	50	5	50	
Zinc	20	5	50	
Boron	100	10	100	
Molybdenu	20	1	10	
Palladium	50	5.0	50	
Sulfur	50	10	100	
Silicon	200	20	200	
Strontium	10	5	50	
Tin	10	10	200	
Titanium	10	1	10	
Tungsten	50	5	50	
Zirconium	10	2	20	
Bismuth	20	2	20	
Lithium	50	5	50	
Phosphorus	50	10	100	
Cerium	100	10	100	

Cerium is being calibrated to monitor IEC factor only.

TABLE 2: ANALYTICAL LINES ON THE SSTRACE			
ELEMENT	WAVELENGTH	PLASMA VIEW	WAVELENGTH RANGE SELECTION
Al	396.1	Radial	High
As	189.0	Axial	Low
Ca	317.9	Radial	High
Fe	259.9	Radial	High
Mg	279.0	Radial	High
Mn	257.610	Axial	High
Pb	220.3	Axial	Low
Se	196.0	Axial	Low
Tl	190.8	Axial	Low
V	292.4	Axial	High
Ag	328.0	Axial	High
Ba	455.4	Radial	High
Be	313.0	Radial	High
Cd	228.8	Axial	Low
Co	228.6	Axial	Low
Cr	267.7	Axial	High
Cu	324.7	Axial	High
K	766.4	Radial	High
Na	589.5	Radial	High
Ni	231.6	Axial	Low
Sb	206.8	Axial	Low
Zn	206.2	Axial	Low
B	208.9	Axial	Low
Mo	202.0	Axial	Low
P	177.4	Axial	Low
S	182.0	Axial	Low
Sr	407.7	Radial	High
Sn	189.9	Axial	Low
Ti	334.9	Axial	High
Si	212.4	Axial	Low
W	207.9	Axial	Low
Zr	339.1	Axial	High
Bi	223.0	Axial	Low
Li	670.7	Radial	High
Ce	404.0	Axial	High

TABLE 3: CALIBRATION STANDARD LEVELS in ug/l		
Element	**STD A (Blank)**	**STD B**
Aluminum	0	80000
Antimony	0	4000
Arsenic	0	4000
Barium	0	4000
Beryllium	0	4000
Cadmium	0	4000
Calcium	0	80000
Chromium	0	4000
Cobalt	0	4000
Copper	0	4000
Iron	0	80000
Lead	0	4000
Magnesium	0	80000
Manganese	0	4000
Nickel	0	4000
Potassium	0	80000
Selenium	0	4000
Silver	0	500
Sodium	0	80000
Thallium	0	4000
Vanadium	0	4000
Zinc	0	4000
Boron	0	4000
Molybdenum	0	4000
Phosphorus	0	4000
Sulfur	0	4000
Silicon	0	**10000**
Strontium	0	4000
Tin	0	4000
Titanium	0	4000
Tungsten	0	4000
Zirconium	0	4000
Bismuth	0	4000
Lithium	0	4000
Cerium	0	4000

TABLE 4: ICV, and CCV LEVELS		
Element	**ICV Suggested Level in ug/l**	**CCV Suggested Level in ug/l**
Aluminum	40000	40000
Antimony	2000	2000
Arsenic	2000	2000
Barium	2000	2000
Beryllium	2000	2000
Cadmium	2000	2000
Calcium	40000	40000
Chromium	2000	2000
Cobalt	2000	2000
Copper	2000	2000
Iron	40000	40000
Lead	2000	2000
Magnesium	40000	40000
Manganese	2000	2000
Nickel	2000	2000
Potassium	40000	40000
Selenium	2000	2000
Silver	250	250
Sodium	40000	40000
Thallium	2000	2000
Vanadium	2000	2000
Zinc	2000	2000
Boron	2000	2000
Molybdenum	2000	2000
Phosphorus	2000	2000
Sulfur	2000	2000
Silicon	5000	5000
Strontium	2000	2000
Tin	2000	2000
Titanium	2000	2000
Tungsten	2000	2000
Zirconium	2000	2000
Bismuth	2000	2000
Lithium	2000	2000
Cerium	N/A	2000

TABLE 5: SUGGESTED CONCENTRATIONS OF METALS IN THE MATRIX SPIKE, SPIKE BLANK AND POST SPIKE FOR AQ, SOIL AND TCLP MATRIX			
ELEMENT	**SOILS FINAL CONCENTRATION IN mg/kg**	**AQUEOUS/SOIL FINAL CONCENTRATION IN µg/l**	
Aluminum	2500	25000	
Antimony	200	2000	
Arsenic	200	2000	
Barium	200	2000	
Beryllium	200	2000	
Cadmium	200	2000	
Calcium	2500	25000	
Chromium	200	2000	
Cobalt	200	2000	
Copper	200	2000	
Iron	2500	25000	
Lead	200	2000	
Magnesium	2500	25000	
Manganese	200	2000	
Nickel	200	2000	
Potassium	2500	25000	
Selenium	200	2000	
Silver	25	250	
Sodium	2500	25000	
Thallium	200	2000	
Vanadium	200	2000	
Zinc	200	2000	
Boron	200	2000	
Molybdenum	200	2000	
Phosphorus	200	2000	
Sulfur	200	2000	
Silicon	500	5000	
Strontium	200	2000	
Tin	200	2000	
Titanium	200	2000	
Tungsten	200	2000	
Zirconium	200	2000	
Bismuth	200	2000	
Lithium	200	2000	
Cerium	N/A	N/A	

TABLE 6: SUGGESTED CONCENTRATIONS OF METALS IN THE QUALITY CONTROL SAMPLE LOW CHECK (CRI or CRIB) SOLUTION	
ELEMENT	**FINAL CONCENTRATION IN µg/l**
Sb	6
As	8
Ba	200
Be	2
Cd	3
Cr	10
Co	50
Cu	10
Pb	3
Mn	15
Ni	10
Se	10
Tl	10
V	50
Zn	20
B	100
Bi	20
Li	50
Mo	20
P	50
Sr	10
S	50
Sn	10
Ti	10
W	50
Zr	10
Ag	5
Si	200
Al	200
Ca	5000
Fe	100
Mg	5000
K	5000
Na	5000
Ce	N/A

TABLE 7: SUGGESTED CONCENTRATIONS OF METALS IN THE QUALITY CONTROL SAMPLE LOW CHECK (CRID) SOLUTION	
Element	**Final Concentration in µg/l**
Sb	
As	3
Ba	4
Be	1
Cd	1
Cr	2
Co	3
Cu	
Pb	
Mn	3
Ni	4
Se	5
Tl	
V	2
Zn	10
B	
Bi	
Li	
Mo	
Pd	
Sr	
S	
Sn	
Ti	
W	
Zr	
Ag	
Si	
Al	100
Ca	1000
Fe	
Mg	100
K	2000
Na	1000
Ce	N/A

TABLE 8: SUGGESTED CONCENTRATIONS OF METALS IN THE QUALITY CONTROL SAMPLE LOW CHECK (CRIA) SOLUTION	
ELEMENT	**FINAL CONCENTRATION IN µg/l**
Sb	20
As	20
Ba	
Be	
Cd	
Cr	
Co	
Cu	
Pb	20
Mn	
Ni	
Se	20
Tl	
V	
Zn	
B	
Bi	
Li	
Mo	
Pd	
Sr	
S	
Sn	200
Ti	
W	
Zr	
Ag	
Si	
Al	500
Ca	2000
Fe	500
Mg	2000
K	
Na	
Ce	

Solution shown in Table 8 is not the part of the routine run but can be used as needed for higher detection limit.

TABLE 9: INTERNAL STANDARDS				
ELEMENTS	**Y 3600**	**Y 3710**	**Y 2243**	**In 2306**
Sb			X	
As			X	
Ba		X		
Be		X		
Cd			X	
Cr	X			
Co				X
Cu	X			
Pb				X
Mn	X			
Ni				X
Se			X	
Tl				X
V	X			
Zn			X	
B			X	
Bi			X	
Li		X		
Mo			X	
P			X	
Sr		X		
S			X	
Sn			X	
Ti	X			
W			X	
Zr	X			
Ag	X			
Si			X	
Al		X		
Ca		X		
Fe		X		
Mg		X		
K		X		
Na		X		
Ce	X			

TABLE 10: PREPARATION AND CONCENTRATION OF ICP DAILY CALIBRATION AND INTERNAL STANDARD

Standard Name	Elements	Vendor Name	Item Name (for multi-elements)	Stock Lot #	Expiration Date	Acid Matrix	Acid Manufacturer	Acid Lot #	Stock Conc. (mg/l)	Vol. Added (ml)	Total Vol. (ml)	Std Conc. (mg/l)	Exp. Date	Date	Analyst (Initials)
MA-_____ STDA	None	N/A		N/A	N/A	__% HNO3 __% HCl			0.000	0.000	1000	0.000			
MA-_____ STDA	None	N/A		N/A	N/A	__% HNO3 __% HCl			0.000	0.000	1000	0.000			
MA-_____ STDA	None	N/A		N/A	N/A	__% HNO3 __% HCl			0.000	0.000	1000	0.000			
MA-_____ STDA	None	N/A		N/A	N/A	__% HNO3 __% HCl			0.000	0.000	1000	0.000			
MA-_____ STDA	None	N/A		N/A	N/A	__% HNO3 __% HCl			0.000	0.000	1000	0.000			
MA-_____ - STDB	B,Ba,Be,Cd,Co, Cr3,Cu,Mn,Ni,P, Pb,Se,Sr,TL,V, Zn	Inorganic Ventures	Accutest-13 REV1			__% HNO3 __% HCl			1000	0.80	200	4.00			
	As,Mo,Sb,Sn,Ti, W,Zr	Inorganic Ventures	Accutest-14 REV1						1000	0.80		4.00			
	Ag	In house							125	0.80		0.50			
	Bi								1000	0.80		4.00			
	Li								1000	0.80		4.00			
	S								1000	0.80		4.00			
	Si								1000	2.00		10.00			
	Al,Na,K,Fe,Mg, Ca	Inorganic Ventures	Metals Mix						5000	3.20		80.00			
	Ce								1000	0.80		4.00			
MA-_____ - IS	Y					__% HNO3 __% HCl			10000	2.00	2000	10.00			
	Cs								10000	40.00		200.00			
	In								10000	10.00		50.00			
MA-_____ - IS	Y					__% HNO3 __% HCl			10000	2.00	2000	10.00			
	Cs								10000	40.00		200.00			
	In								10000	10.00		50.00			

TABLE 11: PREPARATION AND CONCENTRATION OF CCV

Standard Name	Elements	Vendor Name	Item Name (for multi-elements)	Stock Lot #	Expiration Date	Acid Matrix	Acid Manufacturer	Acid Lot #	Stock Conc. (mg/l)	Vol. Added (ml)	Total Vol. (ml)	Std Conc. (mg/l)	Exp. Date	Date	Analyst (Initials)
MA-_____ - CCV	B,Ba,Be,Cd,Co, Cr3,Cu,Mn,Ni,P, Pb,Se,Sr,TL,V, Zn	Inorganic Ventures				__% HNO3 __% HCl			1000	2.00		2.00			
	As,Mo,Sb,Sn,Ti, W,Zr	Inorganic Ventures							1000	2.00		2.00			
	Ag	In house							125	2.00		0.25			
	Bi								1000	2.00		2.00			
	Li								1000	2.00		2.00			
	S								1000	2.00		2.00			
	Si								1000	5.00		5.00			
	Al,Na,K,Fe,Mg, Ca	Inorganic Ventures	Mineral Mix						5000	8.00		40.00			
	Ce								1000	2.00		2.00			

TABLE 12: PREPARATION AND CONCENTRATION OF ICV

Standard Name	Elements	Vendor Name	Name (for multi-elements)	Stock Lot #	Exp. Date	Acid Matrix	Acid Manu-facturer	Acid Lot #	Stock Conc. (mg/l)	Vol. Added (ml)	Total Vol. (ml)	Std Conc (mg/l)	Exp. Date	Analyst (Initials)	Date
	Ba								1000	2.0	1000	2.00			
	Be								1000	2.0		2.00			
	Cd								1000	2.0		2.00			
	Co								1000	2.0		2.00			
	Cr								1000	2.0		2.00			
	Cu								1000	2.0		2.00			
	Mn								1000	2.0		2.00			
	Ni								1000	2.0		2.00			
	V								1000	2.0		2.00			
	Zn								1000	2.0		2.00			
	As								1000	2.0		2.00			
	TL								1000	2.0		2.00			
	Pb								1000	2.0		2.00			
	Se								1000	2.0		2.00			
	Sb								1000	2.0		2.00			
	B								1000	2.0		2.00			
	Mo								1000	2.0		2.00			
	Sn								1000	2.0		2.00			
	Sr								1000	2.0		2.00			
	Ti								1000	2.0		2.00			
	W								1000	2.0		2.00			
	Zr								1000	2.0		2.00			
	S								1000	2.0		2.00			
	Bi								1000	2.0		2.00			
	Li								1000	2.0		2.00			
	P								1000	2.0		2.00			
	Ag								1000	0.250		0.25			
	Si								1000	5.00		5.00			
	Al								10000	4.0		40.00			
	Ca								10000	4.0		40.00			
	Fe								10000	4.0		40.00			
	Mg								10000	4.0		40.00			
	K								10000	4.0		40.00			
	Na								10000	4.0		40.00			

TABLE 13: PREPARATION AND CONCENTRATION OF ICSA AND ICSAB SOLUTIONS.

ICSA:

Standard Name	Elements	Vendor Name	Item Name (for multi-elements)	Stock Lot #	Expiration Date	Acid Matrix	Acid Manufacturer	Acid Lot #	Stock Conc. (mg/l)	Vol. Added (ml)	Total Vol. (ml)	Std Conc. (mg/l)	Expiration Date	Analyst (Initials)	Date
MA-___-___ - ICSA	Mg		N/A			___% Nitric ___% HCl			10000	50.0	1000	500			
	Al		N/A						10000	50.0	1000	500			
	Ca		N/A						10000	40.0	1000	400			
	Fe		N/A						10000	20.0	1000	200			

ICSAB:

Standard Name	Elements	Vendor Name	Item Name (for multi-elements)	Stock Lot #	Expiration Date	Acid Matrix	Acid Manufacturer	Acid Lot #	Stock Conc. (mg/l)	Vol. Added (ml)	Total Vol. (ml)	Std Conc. (mg/l)	Expiration Date	Analyst (Initials)	Date
MA-___-___ - ICSAB	Ag,Cd,Ni,Pb,Zn		CLP ILM 03.0 Analytes B			___% HNO3 ___% HCl			100	10	1000	1.00			
	Ba,Be,Cr,Co,Cu,Mn,V								50	10	1000	0.50			
	Al								10000	50.0	1000	500			
	Ca								10000	40.0	1000	400			
	Fe								10000	20.0	1000	200			
	Mg								10000	50.0	1000	500			
	Sb								1000	1.0	1000	1.00			
	As								1000	1.0	1000	1.00			
	Se								1000	1.0	1000	1.00			
	Tl								1000	1.0	1000	1.00			
	Mo								1000	0.5	1000	0.50			
	W								1000	0.5	1000	0.50			
	Zr								1000	0.5	1000	0.50			
	B								1000	0.5	1000	0.50			
	Sr								1000	0.5	1000	0.50			
	Sn								1000	0.5	1000	0.50			
	Ti								1000	0.5	1000	0.50			
	Si								1000	0.5	1000	0.50			
	S								1000	0.5	1000	0.50			
	Bi								1000	0.5	1000	0.50			
	Li								1000	0.5	1000	0.50			
	P								1000	0.5	1000	0.50			

TABLE 14: PREPARATION AND CONCENTRATION OF HIGH STANDARD

Standard Name	Elements	Vendor Name	Item Name (for multi-elements)	Stock Lot #	Expira-tion Date	Acid Matrix	Acid Manu-facturer	Acid Lot #	Stock Conc. (mg/l)	Vol. Added (ml)	Total Vol. (ml)	Std Conc. (mg/l)	Exp. Date	Date	Analyst (Initials)
MA-_____ - HSTD (Regular)	B,Ba,Be,Cd,Co, Cr3,Cu,Mn,Ni,P, Pb,Se,Sr,TL,V, Zn	Inorganic Ventures	Accutest-13 REV1			__% HNO3 __% HCl			1000	8.00	1000	8.00			
	As,Mo,Sb,Sn,Ti, W,Zr	Inorganic Ventures	Accutest-14 REV1						1000	8.00		8.00			
	Ag	In house							125	5.00		0.625			
	Bi								1000	8.00		8.00			
	Li								1000	8.00		8.00			
	S								10000	10.00		100.00			
	Si								10000	2.50		25.00			

Standard Name	Elements	Vendor Name	Item Name (for multi-elements)	Stock Lot #	Expira-tion Date	Acid Matrix	Acid Manu-facturer	Acid Lot #	Stock Conc. (mg/l)	Vol. Added (ml)	Total Vol. (ml)	Std Conc. (mg/l)	Exp. Date	Date	Analyst (Initials)
MA-_____ - HSTD (Minerals)	Al					__% HNO3 __% HCl			10000	30.00	1000	300.00			
	Mg								10000	30.00		300.00			
	K								10000	20.00		200.000			
	Na								10000	20.00		200.00			
	Ca								10000	20.00		200.00			
	Fe								10000	20.00		200.00			

TABLE 15: PREPARATON AND CONCENTRATION OF CRI SOLUTION

Standard Name	Elements	Vendor Name	Item Name (for multi-elements)	Stock Lot #	Expira-tion Date	Acid Matrix	Acid Manu-facturer	Acid Lot #	Stock Conc. (mg/l)	Vol. Added (ml)	Total Vol. (ml)	Std Conc. (mg/l)	Expira-tion Date	Analyst (Initials)	Date
	Sb					___% HNO3			6.00			0.006			
	Mo	Inorganic Ventures	ACCUTEST-20A			___% HCl			20.00			0.020			
	Sn								10.00	**1.00**		0.010			
	Ti								10.00			0.010			
	W								50.00			0.050			
	Zr								10.00			0.010			
	Al								200.00			0.200			
	As								8.00			0.008			
	Ba								200			0.200			
	Be	Inorganic Ventures	ACCUTEST-20B						2.00			0.002			
	B								100.00			0.100			
	Cd								3.00			0.003			
	Ca								5000			5.000			
	Cr								10.0	**1.00**	**1000**	0.010			
	Co								50.00			0.050			
	Cu								10.0			0.010			
	Fe								100.0			0.100			
MA-_____ - CRI	Pb								3.0			0.003			
	Mg								5000			5.000			
	Mn								15.0			0.015			
	Ni								10.0			0.010			
	P								50.0			0.050			
	K								5000			5.000			
	Se								10.0			0.010			
	Na								5000.0			5.000			
	Sr								10			0.010			
	TL								10			0.010			
	V								50			0.050			
	Zn								20			0.020			
	Bi	In house	Intermediate						20	1.00		0.020			
	Li	In house	Intermediate						50	1.00		0.05			
	S	In house	Intermediate						50	1.00		0.05			
	Zr	In house	Intermediate						10	1.00		0.01			
	Si								1000	0.20		0.20			
	Ag	In house	Intermediate						10	0.50		0.005			

TABLE 16: PREPARATION AND CONCENTRATION OF CRID SOLUTION

Standard Name	Elements	Vendor Name	Item Name (for multi-elements)	Stock Lot #	Expiration Date	Acid Matrix	Acid Manufacturer	Acid Lot #	Stock Conc. (mg/l)	Vol. Added (ml)	Total Vol. (ml)	Std Conc. (mg/l)	Expiration Date	Analyst (Initials)	Date
	Al					___% HNO3			100.00			0.100			
	As	Inorganic Ventures	ACCUTEST-21			___% HCl			3.00			0.003			
	BA								4.00	1.00	1000	0.004			
	Be								1.00			0.001			
	Cd								1.00			0.001			
MA-_____ - CRID	Ca								1000.00			1.000			
	Cr								2.00			0.002			
	Co								3.00			0.003			
	Mg								100			0.100			
	Mn								3.00			0.003			
	Ni								4.00			0.004			
	K								2000.00			2.000			
	Se								5			0.005			
	Na								1000.0			1.000			
	TL								2.00			0.002			
	V								2.0			0.002			
	ZN								10.0			0.010			

TABLE 17: PREPARATION AND CONCENTRATION OF POST SPIKE SOLUTION

SGS					Date		Analyst		MP #	
Post spike for ICP Method 6010D					**QC sample #**					
Spiking solution	Elements	Spike added (Y/N)	Vendor	Intermediate Lot #	Exp. Date	Conc. (mg/l)	Amt of Spike added (ml)	Digestate sample volume (ml)	Final Digestate volume in ml (Spike+ Sample)	Final Conc at the instrument (mg/l)
Mixed ICP intermediate Metals Solution (ACCUTEST-13A-REV1)	Ba,Be,B Cd,Cr,Co,Cu,Pb ,Mn,Ni,P,Se,Sr, TL,V,Zn					200	0.2			2
Mixed ICP intermediate Metals Solution (ACCUTEST-14A-REV1)	Sb,As,Mo,Sn,Ti, W,Zr					200	0.2		20	2
Ag Spike Intermediate	Ag					20	0.25			0.25
Metals Mix	Ca,Al,Fe,Mg,K, Na					5000	0.1			25
S spike	S					200	0.2			2
Bi Spike	Bi					200	0.2			2
Li Spike	Li					200	0.2			2
Si spike	Si					1000	0.1			5

For preparations of post spike Intermediate solutions follow the ICP Intermediate preparation book.

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
reference	Added revision and year
4.0	Updated MDL definition
6.1	Revised preservation temperature
12.2	Updated MDL procedure requirement

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By

END OF DOCUMENT

LAB SUPERVISOR: _____

QA OFFICER: _____ Olga Y. Ajaimo

EFFECTIVE DATE: 3-15-2021

TITLE: DIGESTION OF NON-POTABLE WATERS FOR ICP OR ICP-MS ANALYSIS.
REFERENCES: SW846 3010A
REVISED SECTIONS: 6.1.2

1.0 SCOPE & APPLICATION

1.1 This method is applicable for the digestion of aqueous samples, TCLP extracts, and wastes that contain small amounts of suspended solids. After digestion, the samples can be analyzed by ICP or by ICP-MS. This digestion method is based on SW846 3010A.

2.0 SUMMARY

2.1 Samples for metals analysis are digested on a hot plate or in a digestion block at 90 to 95° C to solubilize the metals before analysis. Nitric and hydrochloric acids are used for digestion.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. See determinative method.

3.2 Method Detection Limit. MDLs must be established using a solution spiked at approximately 3 to 5 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of three replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs must be determined approximately once per year for frequently analyzed parameters.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a batch. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

SPIKE BLANK Digest and analyze a spike blank with each set of samples. A minimum of one spike blank is required for every 20 samples. Assess laboratory performance against the control limits specified in the SOP. In house limits must also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses) If the Spike blank is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the spike blank recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be

reported with no flag. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent must be used.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample must be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results must be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and must be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent must be used.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

$$\frac{(|\text{MS Result} - \text{MSD Result}|) \times 100}{(\text{MS Result} + \text{MSD Result})/2} = \text{MSD RPD}$$

METHOD BLANK: The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLS). The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLs are determined approximately once per year for frequently analyzed parameters.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

REFERENCE MATERIAL: A material that containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Laboratory Safety Manual which includes the use of safety glasses and lab coats. All digestions must be done in a hood. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Expose to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemical specified in this method. A reference file of date handling sheets must be made available to all personnel involved in these analyses.

6.0 COLLECTION, PRESERVATION, & HOLDING TIMES

6.1 All samples must be preserved with nitric acid at the time of collection to a pH of < 2.

6.1.1 Samples received without preservation must be preserved following the specifications in the SGS QSM.

6.1.2 It is recommended that an additional unpreserved aliquot be taken for samples which require total sulfur if significant sulfide is suspected to be present. (The additional of acid may promote loss of sulfide from the sample.) See section 10.3.5 for handling of the sulfur aliquot.

6.2 All samples must be digested and analyzed within 6 months of the time of collection.

7.0 APPARATUS AND MATERIALS

7.1 The apparatus needed for this digestion procedure are listed below.

7.2 Digestion block. Temperature adjustable and designed to hold sample digestion tubes and capable of maintaining temperatures from 90 to 95°C.

7.3 Thermometers calibrated with NIST traceable thermometers. To be used to monitor digestion temperatures.

7.4 Sample digestion tubes and ribbed watch glasses.

7.4.1 If sample digestion tubes are used to measure initial and final volumes, then they must be calibrated following the procedure outlined in EMP203, the digestion tube calibration SOP.

7.5 Automatic pipeter bottles.

7.6 50 ml volumetric flasks or 50 ml TD volumetric cylinders (Class A).

7.6.1 All glassware must be washed with soap and tap water and then soaked in a 10% nitric acid bath for several hours. It must then be rinsed at least 3 times with distilled, deionized water.

7.7 Glass funnels.

7.8 Whatman #41 filter paper or equivalent.

7.9 Volumetric pipets, class A.

7.10 pH paper.

7.11 Auto pipet

8.0 STANDARDS & REAGENTS

8.1 All chemicals listed below are reagent grade unless otherwise specified. Deionized water must be used whenever water is required.

8.2 Hydrochloric acid. Baker instra-analyzed or equivalent.

8.3 Nitric Acid. Baker instra-analyzed or equivalent.

8.4 30% Hydrogen peroxide

8.5 Metals Spiking Solutions. All metals spiking solutions must be made up in a solution of 2 % nitric acid following the procedures outlined in EMP202-Spiking Procedure. This can be purchased as a mixture from outside vendor.

8.6 Hydrogen Peroxide, 30%. (Used only if sulfur is an analyte of interest).

9.0 INTERFERENCES

9.1 Organics in a matrix may cause interferences if the sample is not digested rigorously enough. In addition, high levels of acids in the final digestate may cause interferences in the analysis. Both of these interferences can be avoided by choosing the appropriate digestion method and by bringing the sample to an appropriate final volume. For a discussion of other interferences, refer to specific analytical methods.

10.0 PROCEDURE

10.1 Below is the procedure to be followed for the digestion of aqueous samples prior to ICP or ICP-MS analysis.

10.2 Before starting the analysis, check the initial pH of the sample with pH paper and verify that it is < 2. Below is procedure for the checking pH and preservation of metals samples when pH is NOT <2. Add a small amount of concentrated nitric acid (~2mls) dropwise to the sample and mix. Using a pipet tip, place a small amount of sample on a piece of pH paper or pour directly on the pH paper at the sink to confirm that the pH is <2. Document the date and time of the pH adjustment and the lot number of the nitric acid used for the preservation on the preservation log. Mark the date and time of preservation on the bottle to ensure that the analysis is not started for 24 hours from the time of preservation.

10.2.1 For highly basic or buffered samples, where more than a few drops of acid are required, adjust an aliquot of the sample rather than the whole amount and record the amount of acid used for the adjustment.

10.2.2 Samples for TCLP matrix or leachate spikes will be received unpreserved from the TCLP leaching area. These must be aliquoted, spiked, and preserved on receipt in the metals prep area.

10.2.2.1 For handling leachate oil samples refer to the SOP EMP073.

10.2.3 For some clients, additional documentation of the pH adjustment is necessary. For all samples from Ohio, the pH adjustment must be noted in the case narrative or conformance summary for the samples.

10.3 Measure out 50 ml of each nitric preserved sample into a labeled digestion tube. The sample may be measured by using a graduated cylinder or by using a calibrated digestion tube. Make sure that the sample identification is accurately recorded with the digestion tube numbers on the sample digestion log. In addition to the samples, a Spike Blank and a Method Blank must be set up with each batch of 20 samples or less. A Matrix Spike and a Matrix Spike Duplicate (or Matrix Duplicate) must be set up with each batch of 20 samples. Matrix Spike Duplicates are normally used unless otherwise specified by client requirements. Check with the metals supervisor for spiking levels to use for the matrix spikes and the spike blank.

10.3.1 For the method blank, add 50 ml of deionized water to the digestion tube.

10.3.2 For the Blank Spike, add Spike solution to 50 ml of DI water in to the digestion tube. Refer to the metals spiking solution SOP, EMP202.

10.3.3 For the matrix spike and matrix spike duplicates, add the spiking solution directly to the 50 ml of sample in the designated tubes. Refer to the metals spiking solution SOP, EMP202, for information on the preparation and amounts of spiking solution required.

10.3.4 TCLP blank spikes must be prepared using 50 ml of the appropriate TCLPE extraction fluid.

10.3.5 For samples to be tested for sulfur, where sulfide is suspected to be present, measure out 50.0 ml of unpreserved sample into a labeled digestion tube. Add 5.0 ml of 30% hydrogen peroxide and let the samples sit for 30 minutes. Then add concentrated nitric acid drop wise to a pH of <2. Record the lots added. All associated quality control (method blank, spike blank, matrix spike, and matrix spike duplicate) must be treated in the same manner. The spikes must be added before the addition of the hydrogen peroxide and acid. Record these steps on the digestion log.

10.3.5.1 If other metals are to be tested from the treated sample from 10.3.5, then it must sit for 24 hours before continuing the digestion. It is recommended that a nitric acid preserved bottle from the field be used for all other metal's digestions.

10.4 Add 1.5 ml of concentrated nitric acid to all quality control and samples.

10.5 Place the labeled tubes into a digestion block and cover with watch glasses. Heat the samples at 90 to 95°C until they come to a gentle reflux and then continue to heat the samples until they are evaporated to near dryness, for example 10 ml or less. After the heating is complete, allow the samples to cool.

10.5.1 Watch glasses may be removed if necessary, to allow for faster volume reduction, but must be left on for the maximum time possible to limit contamination.

10.6 Add an additional 1.5 ml of concentrated nitric acid to all quality control and samples. Continue heating the samples at a gentle reflux until the sample is completely digested. (More acid may be added as necessary to complete the digestion.)

10.6.1 Signs of a complete digestion are if the digestate is light in color and/or if the appearance of the sample does not change with continued refluxing.

10.7 Add 5 ml of 1:1 HCl to each sample and reflux for an additional 15 minutes.

10.8 Wash down the distillation tube walls and watch glass with DI water. Bring the sample to a final volume of 50 ml with deionized water. The sample is now ready for analysis by ICP.

10.8.1 If the sample contains particulate, it can be filtered through Whatman # 41 filter paper (or equivalent) before analysis. If any samples in a batch are filtered, then all of the associated quality control must also be filtered in the same manner.

10.8.2 For ICP-MS analysis, the digestate is normally further diluted at the instrument before analysis (at least by a factor of 2) and the dilution factor must be added to the instrument file.

11.0 QUALITY ASSURANCE

11.1 A sample batch is defined as a maximum of 20 field samples in a preparation batch over a time period of 24 hours. A matrix spike/matrix spike duplicate, matrix spikes and/or duplicate is required every 20 samples.

11.2 For each digestion batch of 20 samples or less a spike blank and a method blank is prepared.

11.3 For every 20 samples, digest a matrix spike/matrix spike duplicate pair instead of a matrix spike/ duplicate pair unless otherwise requested by a client.

11.4 Refer to the analytical methods SOPs for additional information on method quality control.

12.0 DOCUMENTATION

12.1 All digestion information must be entered on a digestion log. The information required includes the sample identification, the initial sample volume, the final sample volume, the initial sample pH, the acids used (including both amount and lot number), the spikes used, and the digestion times and temperatures, and the thermometer identification. Both the corrected and uncorrected temperatures must be recorded. If filtration is done, the filter type and lot must also be recorded.

12.2 The analyst must write additional information such as unusual sample characteristics in the Comments section. All spiking solution information must be entered in the metals spiking solution notebook.

13.0 DATA REVIEW & REPORTING

13.1 The prep analyst is responsible for updating the samples to SCH status in the LIMS system and for entering the prep information into the LIMS. This may be done manually or electronically. When the prep information is in the LIMS, the completed paperwork must be turned into the metals supervisor for review.

13.2 The supervisor or a metals analyst reviews the preparation information and approves the data in the LIMS system.

13.3 The original paperwork is submitted to the report generation department for filing.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non-hazardous aqueous wastes.

14.2.2 Hazardous aqueous wastes.

14.2.3 Chlorinated organic solvents.

14.2.4 Non-chlorinated organic solvents.

14.2.5 Hazardous solid wastes.

14.2.6 Non-hazardous solid wastes.

15.0 ADDITIONAL REFERENCES

15.1 Refer to the ICP and ICPMS analytical SOP's, the spiking procedure SOP (EMP202), and the digestion tube calibration SOP (EMP203).

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
6.1.2	Revised reference to the section for handling sulfur aliquot

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
19	8/22/2019	Maria Ruschke
20	01/04/2021	Rakesh Pathak
21	03/10/2021	Olga Azarian

END OF DOCUMENT

LAB SUPERVISOR: Natasha Vera

QA OFFICER: Olga G. Gaiaan

EFFECTIVE DATE: 3-20-2024

TITLE: DIGESTION OF SOILS FOR ICP AND ICP-MS ANALYSIS
REFERENCES: SW846 3050B (Revision 2, December 1996)
REVISED SECTIONS: 8.7

1.0 SCOPE AND APPLICATION

1.1 This method is applicable for the digestion of sediments, soils, sludges, solid wastes, and wipes. After digestion, the samples can be analyzed by ICAP or by ICP-MS (or by graphite furnace AA for antimony). This digestion method is based upon SW846 method 3050B, Revision 2, 12/96.

2.0 SUMMARY

2.1 For the digestion of samples, a representative 1-2 gram (wet weight) or 1 gram (dry weight) of sample is
digested with repeated additions of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2). Then hydrochloric acid (HCl) is added to the initial digestate and the sample is refluxed. In an optional step to increase the solubility of some metals, this digestate is filtered and the filter paper and residues are rinsed, first with hot HCl and then hot reagent water. Filter paper and residue are returned to the digestion flask, refluxed with additional HCl and then filtered again. The digestate is then diluted to a final volume of 100 Ml.

2.2 In method 3050B, the final HCl addition is not included for ICP-MS digestates as chloride is interference for several elements on ICP-MS. However, the HCl is necessary for good solubility of many elements. HCl interferences at the ICP-MS can normally be addressed with corrections at the ICP-MS. Therefor it is recommended that HCl be added for ICP-MS digestions by this method unless otherwise directed by the lab supervisor or manager.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 See determinative method.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed, and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

SPIKE BLANK OR LAB CONTROL SAMPLE. Digest a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. Assess laboratory performance against the control limits specified in the SOP. In house

limits must also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent must be used.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample must be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results must be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and must be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent must be used.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

MATRIX SPIKE DUPLICATES: Intra laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

$$\frac{(|\text{MS Result} - \text{MSD Result}|) \times 100}{(\text{MS Result} + \text{MSD Result})/2} = \text{MSD RPD}$$

METHOD BLANK: The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLS). The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is distinguishable form method blank results.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Plan and Personal Protection Policy, which include the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

6.0 PRESERVATION & HOLDING TIME

6.1 Non-aqueous samples must be refrigerated at the time of receipt.

6.2 All samples must be digested and analyzed within 6 months of the time of collection.

7.0 INTERFERENCES

7.1 Sludge and soil samples can contain diverse matrix types, which may contain a variety of interferences. Spiked samples can be used to determine if these interferences are adequately treated in the digestion process. For a discussion of other interferences, refer to specific analytical methods.

8.0 APPARATUS

8.1 The apparatus needed for this digestion procedure are listed below

8.2 Digestion block. Temperature adjustable and designed to hold sample digestion tubes and capable of maintaining temperatures from 90 to 95°C.

8.3 Thermometers calibrated with NIST traceable thermometers. To be used to monitor digestion temperatures.

8.4 Sample digestion tubes and ribbed watch glasses.

8.5 Automatic pipeter bottles.

8.6 100 ml volumetric flasks.

8.7 Glass funnels, which can be reused after cleaning in accordance with EQA012 for glassware cleaning.

8.8 Fisher Q8 filter paper.

8.9 Top loader balance.

8.10 Volumetric Pipets, class A.

8.11 Disposable Wood Spatulas

8.12 Ceramic Mixing Bowl.

8.13 Auto pipettes

9.0 REAGENTS

9.1 All chemicals listed below are reagent grade unless otherwise specified. Deionized water must be used whenever water is required.

9.2 Hydrochloric acid. Baker Instra-analyzed or equivalent.

9.3 Nitric Acid. Baker Instra-analyzed or equivalent.

9.4 Hydrogen Peroxide, 30 %.

9.5 Metals Spiking Solutions. All metals spiking solutions must be made up in a solution of 2 % nitric acid as described in Table 1. Mixture of Spike solution can be purchased from the outside vendor (eg. Inorganic ventures/Agilent, Absolute etc.). Use volumetric glassware and pipets or autopipets. Check with the metal's supervisor for additional information. Different levels of spiking solutions may be used as specified by the area supervisor.

9.5.1 The expiration date for the spiking solution is defined as the earliest date of any element or compound in that solution.

9.6 Teflon Chips

9.7 Solid Lab Control (Mixture of Metals analytes in Soil Matrix). This would be commercially purchased.

10.0 PROCEDURE: Using Q8 (or equivalent) Filter paper

10.1 Weigh out an amount of wet sample equivalent to 1 g of dry sample into a numbered digestion tube. Label each digestion tube with the complete sample number, the MP number, Date, and the number corresponding to the flask used for filtration (see section 10.11). The sample must be weighed out using a top loader balance and the weights must be recorded to two places past the decimal. Make sure that the sample identification is accurately recorded with the digestion tube numbers on the sample digestion log.

10.1.1 Make sure that the sample has been thoroughly mixed before weighing out the representative sample. Discard rocks, sticks, etc. from the sample. (Refer to the SOP EQA042 for proper sample aliquot procedures). All homogenization and sample handling must be done with wooden spatulas and ceramic (or other non-metal) bowls.

10.1.2 If the sample has low percent solids, a larger sample size may be used to obtain a weight approximately equivalent to 1 g of dry sample. Check with the metals supervisor for additional direction with sampled with low percent solids or unusual matrices.

10.1.3 If the sample is a wipe, weighing is not necessary. Transfer the entire wipe into the labeled digestion tube and proceed with the digestion following steps 10.2 through 10.10. Extra wipes must be supplied by the client for the matrix spike or duplicate.

10.1.4 If the sample is leachate oil, measure 1ml of the sample and follow the steps starting at section 10.4. Leachate oil samples are handled using soil procedure on a volume basis.

10.2 In addition to the samples, a spike blank or a lab control and a method blank must be set up with each batch of 20 samples or less. A matrix spike, a matrix spike duplicate or a duplicate must be set up with each batch of 20 samples. Matrix spike duplicates are normally used unless otherwise specified by client requirements. Refer to Table 1 for spiking levels to use for each MS and Spike Blank.

10.2.1 For the method blank and spike blank, instead of weighing out soil add approximately 1 g of Teflon chips to the digestion tube. Add the spiking solution to the blank spike after the chips are in the tube.

10.2.2 For some clients, a solid lab control is required rather than a blank spike. In that case, the solid lab control must be weighed out in the same manner as a sample.

10.2.3 For the matrix spike and matrix spike duplicate, refer to the metals spiking SOP, EMP202, for information on the preparation and amount of spiking solution require to be added to the sample in the digestion tube.

10.2.4 For additional details on spiking solutions and preparation, refer to the metals spiking sop, EMP202.

10.2.5 For leachate oil QC samples use 1ml of leachate and follow the steps starting at section 10.4

10.3 For samples to be tested for sulfur, where sulfide is suspected to be, add 2.0 ml of 30% hydrogen peroxide and let the samples sit for 30 minutes. All associated quality control (method blank, spike blank or lab control, matrix spike, and matrix spike duplicate) must be treated in the same manner. The spikes must be added before the addition of the hydrogen peroxide and acid. Record these steps on the digestion log. Proceed to step 10.5

10.4 Add 10 ml of 1:1 nitric acid to all Quality Control and samples.

10.5 Place the numbered tubes into a digestion block and cover with watch glasses. Heat the samples at 90 to 95°C until they come to a gentle reflux and then continue to heat the samples for an additional 10 to 15 minutes. Do not allow the samples to boil. After the heating is complete, allow the samples to cool.

10.6 Add an additional 5ml of concentrated nitric acid to all quality control and samples. Heat the samples at a gentle reflux for an additional 30 minutes. Do not allow the volume to be reduced to below 5 ml. Cool.

10.6.1 If brown fumes are generated during this digestion, add an additional 5ml aliquot of concentrated nitric acid and heat for 30 more minutes. Repeat this process until no more brown fumes are generated.

10.7 Continue the digestion at 90 to 95ºC until the volume is reduced to 5 ml or for a period of 2 hours. Do not let the samples boil at any point during the digestion process.

10.8 Add 2 ml of water and 3 ml of 30 % hydrogen peroxide to each sample and reflux until the effervescence subsides. Cool.

10.8.1 The 30% hydrogen peroxide may be added in smaller or larger initial aliquots depending on the sample characteristics. If sample effervescence is suspected to be a problem, smaller aliquots must be used.

10.9 Continue to add 30 % hydrogen peroxide in 1ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. NOTE: Do not add more than a total of 10 ml of 30 % hydrogen peroxide. Continue the digestion at 90 to 95ºC until the volume is reduced to 5 ml or for a period of 2 hours.

10.9.1 For the batch that needs total sulfur, do not add more than a total of 8 ml of 30% hydrogen peroxide at this stage of the digestion because 2 ml is added prior to the digestion.

10.10 Add 10 ml of concentrated HCl and reflux for an additional 15 minutes.

10.10.1 HCl may be omitted for some ICP-MS digestions for limited elements (As, Be, Cd, Cr, Co, Fe, Pb, Mo, Se, and Tl) at the discretion of the area supervisor or manager. However, in normal circumstances it must be added. Refer to section 2.2.

10.11 Filter the digestate through Fisher Brand Q8 filter paper (or equivalent) into 100 ml volumetric flasks and be sure to label all the flasks with the numerical value assigned to the digestion tube containing the corresponding sample (ex. 1,2,3). Make sure to rinse the digestion tubes and the filter paper well with deionized water. Dilute to volume of 100 ml with deionized water and mix well. The sample is now ready for analysis.

10.11.1 For ICP-MS analysis, the digestate must be further diluted at the instrument before analysis (normally at least by a factor of 2 to 5) and the dilution factor must be added to the instrument file.

11.0 QUALITY ASSURANCE

11.1 For each digestion batch of 20 samples or less, a lab control or a spike blank and a method blank must be prepared. Solid lab controls are required for some clients. Check with the metals supervisor for additional information.

11.2 For every 20 samples, a matrix spike, a matrix spike duplicate or a duplicate must be prepared. Matrix spike duplicates are normally used unless otherwise specified by the client requirements.

11.3 If a batch is a running batch, samples can be added to the batch (along with a method blank and spike blank or lab control) up to a maximum of 2 weeks.

11.3.1 Running batches are not allowed for all clients. Check with the metals supervisor for additional information.

11.4 Refer to the analytical methods SOPs for additional information on method quality control.

12.0 DOCUMENTATION

12.1 All digestion information must be entered on a digestion log. The information required includes the sample identification, the initial sample weight, the final sample volume, the acids used (including both amount and lot number), the spikes used, and the digestion times, temperatures, and the thermometer identification. Both the corrected and uncorrected temperature must be recorded.

12.2 The analyst must write additional information such as unusual sample characteristics in the Comments section. All spiking solution information must be entered in the metals spiking solution notebook.

12.3 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

12.4 Certificates of analysis for all primary stocks must be kept on file. If a certificate is received in the lab, give the original to the area supervisor for submission for filing with the QA department.

13.0 DATA REVIEW & REPORTING

13.1 The prep analyst is responsible for updating the samples to SCH status in the LIMS system and for entering the prep information into the LIMS. This may be done manually or electronically.

When the prep information is in the LIMS, the completed paperwork must be turned into the metals supervisor for review.

13.2 The supervisor or a metals analyst reviews the preparation information and approves the data in the LIMS system.

13.3 The original paperwork is submitted to the report generation department for filing.

13.4 Supervisory (or peer) personnel will review and sign all reagent documentation a minimum of once per month.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non-hazardous aqueous wastes.

14.2.2 Hazardous aqueous wastes.

14.2.3 Chlorinated organic solvents.

14.2.4 Non-chlorinated organic solvents.

14.2.5 Hazardous solid wastes.

14.2.6 Non-hazardous solid wastes.

15.0 ADDITIONAL REFERENCES

15.1 Refer to the ICP and ICPMS analytical SOP’s and the spiking procedure SOP (EMP202).

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
8.7	Added reference to SOP for glassware cleaning

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
21	8/22/19	Maria Ruschke
22	01/04/21	Rakesh Pathak
23	3/20/2024	Olga Azarian

END OF DOCUMENT

LAB SUPERVISOR: Rak

QA OFFICER: Jonfap.

EFFECTIVE DATE: 03/21/2024

TITLE: ANALYSIS OF PER- AND POLYFLUORINATED ALKYL SUBSTANCES (PFAS) IN AQUEOUS AND SOLID SAMPLES BY LC/MS/MS

REFERENCES: EPA METHOD 1633, January 2024

REVISED SECTIONS: references, 2.1, 15.1, Table 6.

ADDED SECTIONS: NONE

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentrations of select Per- and Polyfluorinated Alkyl Substances (PFAS) in aqueous, solid (soil, sediment, biosolids), and tissue matrices utilizing an HPLC equipped with a tandem mass spectrometer (MS/MS).

1.2 Analytes that may be reported under this method are listed in TABLE 1. Translations between analytes names and acronyms used in EPA 1633 versus the laboratory report and raw data are listed in TABLE 4.

1.3 This method is "performance-based," meaning that modifications may be made without additional EPA review to improve performance (e.g., overcome interferences, or improve the sensitivity, accuracy, or precision of the results) provided that all performance criteria in this method are met.

2.0 SUMMARY OF METHOD

2.1 This method is adapted from EPA Method 1633 for the analysis of environmental water and soil samples. This SOP is not designed to be used to analyze aqueous and solid samples by the laboratory's in-house LCMSMS method.

2.2 Samples are received, stored, and extracted within the appropriate holding times.

2.3 Sample preparation is performed following SGS-Dayton SOP EOP040 and EOP041.

2.4 Samples known to be high in PFAS (such as AFFF or AFFF impacted waters) should be screened by serial dilution and direct injection onto the LC/MS/MS in order to determine the appropriate subsample size. High level water and soil samples require that a smaller sample aliquot be used so that the analytes fall within the instrument calibration range. For definitive analysis AFFF samples must be subcontracted to a laboratory certified for AFFF analysis by QSM 5.4.

2.5 Per- and Polyfluorinated Alkyl Analytes are separated, detected and quantitated using an LC/MS/MS. After HPLC separation and ionization, the specific Perfluorinated compound is isolated in the first mass spectrometer and transferred to a collision cell for fragmentation.

The resulting fragments are introduced into the second mass spectrometer where they are detected and quantified.

2.6 Per- and Polyfluorinated Alkyl Analytes may exist in branched and/or linear form. Fluorotelomer production results in linear isomers only but electrochemical fluorination results in branched and linear isomers. The branched isomers may account for up to 30% of the total analyte. The branched isomers will elute just before the linear isomer. A qualitative branched/linear RT standard with additional branched isomers is used to help establish transition windows.

2.7 Manual integrations are performed in accordance with SOP EQA044.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 The Lower Limit of Quantitation (LLOQ) or Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. LLOQs may vary depending on matrix complications and volumes. LLOQs for this method are 0.002-0.050 ug/l for aqueous samples and 0.2-50 ug/kg for solid samples. Solid matrices are reported on a dry weight basis.

3.2 **MeFOSA, EtFOSA, MeFOSE, and EtFOSE** tend to recover erratically by SPE cartridge. These analytes may also be lost during the evaporative step. Data for these analytes should be reviewed carefully.

3.3 The Method Detection Limit (MDL). Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B, Revision 2. Experimental MDLs must be determined in accordance with SGS SOP EQA075

3.4 Compounds detected at concentrations between the LLOQ and MDL are quantitated and qualified as estimated values and reported with a “J” qualifier. Some program or project specifications may require that no values below the LLOQ be reported.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours whichever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known number of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Low Level Blank Spike (LLBS): An analyte-free matrix spiked with a known number of analyte(s) at 2x LLOQ, processed simultaneously with the samples through all the steps of the analytical procedure. Low-Level Blank Spike Recoveries are used to document laboratory performance at the LLOQ for a given method. This may also be called a Low Level Laboratory Control Sample (LLLCS) or the Low-Level Ongoing Precision And Recovery Standard (LLOPR).

4.4 Extracted Internal Standards (EIS): A standard containing isotopically labelled versions of the native target analytes. These isotopes are usually labelled with C13, d2, or O18 atoms. Isotope Dilution Standards are used to measure the extraction efficiency and to correct the concentrations of the native analytes based on the recovery of their isotopically labelled analogs.

4.5 Field Blank (FB): An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are present in the field environment.

4.6 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and are still considered valid.

4.7 Matrix Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.9 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Laboratory Safety Manual which includes the use of Safety glasses, gloves and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the SDS prior to using any new reagents or solvents.

5.3 Methanol is an inhalation hazard. Use in well ventilated area.

5.4 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory maintains a

current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets is available to all personnel involved in these analyses.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIME

6.1 Collection

6.1.1 Aqueous samples should be collected in 500mL high density polyethylene bottles (HDPE). Caps must not have Teflon liners. Alternate size bottles may be used depending on project requirements. Additional bottles should be provided for solids determination, dilutions, and pre- screening of samples.

6.1.2 All containers must demonstrate to be PFAS-free at or above the MDLs for the target analytes by testing one or more representative containers from each lot.

6.1.3 Solid samples shall be collected in 4oz or 2oz HDPE wide mouth jars. Caps must not have Teflon liners.

6.1.4 The samples must be chilled to ≤ 6°C from the time of collection until arrival at the laboratory.

6.2 Storage

6.2.1 Samples may be stored in the dark at either ≤ 6°C or ≤ -20°C.

6.2.1.1 Issues were observed with MeFOSE, EtFOSE, MeFOSAA and EtFOSAA after 7 days when stored at ≤ 6°C. These issues are more likely to elevate the observed concentrations of other PFAS compounds via the transformation of these precursors if they are present in the sample.

6.2.2 The extracts should be stored in the dark at ≤ 6°C. All extracts must be allowed to come to room temperature and vortexed just prior to transfer to the autosampler vials.

6.3 Holding Time

6.3.1 Aqueous and solid samples must be extracted and analyzed within 28 days of collection if stored at ≤ 6°C.

6.3.2 Aqueous and solid samples must be extracted and analyzed within 90 days of collection if stored at ≤ -20°C.

6.3.3 Leachates must be leached within 28 days of collection and extracted and analyzed within 28 days from leaching date.

7.0 APPARATUS AND MATERIALS

7.1 HPLC – Agilent Technologies 1260 or 1290

7.1.1 Suitable HPLC equipped with an autosampler, pump, and column compartment. System may have a membrane degasser if shown to not adversely affect the analysis.

7.2 MS/MS – Agilent Technologies 6470A or 6495B

7.2.1 LC/MS/MS must be capable of negative ion electrospray ionization near the required flow rate of the HPLC Column. The system must be capable of performing MS/MS to produce unique precursor and product ions for the PFAS method analytes within the specified retention time segments. A minimum of 10 scans across each peak is required to ensure adequate precision.

7.3 Data System – Agilent Technologies Mass Hunter B10.0x

7.3.1 A computer system interfaced to the HPLC/MS/MS that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

7.3.2 The software must allow for the viewing of the specific MS/MS Spectra acquired over the analytical run. Comparisons can then be made between spectra from standards and samples.

7.3.3 Data is archived to a backup server for long term storage.

7.4 Columns: Agilent Poroshell 120 EC C18 2.7um, 100 x 2.1 mm ID or equivalent

7.5 Delay Columns: Agilent Poroshell or Eclipse C18 50 x 4.6 mm ID or equivalent

7.6 Disposable polyethylene transfer pipettes

7.7 15ml Centrifuge tubes

7.8 HDPE or Polypropylene screw cap and autosampler vials

7.9 Volumetric Pipettors and volumetric “plasticware” for dilutions of standards and extracts.

7.10 Class A volumetric flasks.

7.11 HDPE bottles – various sizes, shown to be PFAS free.

8.0 STANDARDS AND REAGENTS

8.1 Acetonitrile – HPLC grade or equivalent (Eluent A)

8.2 Water – HPLC grade or equivalent

8.3 Ammonium Acetate – LCMS grade or equivalent (Replace 2 Years after opening date)

8.4 Ammonium Hydroxide – Fisher A669-212 or equivalent (28-30% Aqueous Ammonia(replace 2 years after opening date).

8.5 Eluent A - Acetonitrile

8.6 Eluent B – 2mM Ammonium Acetate in 95:5 Water: Acetonitrile
Dissolve 0.154 grams of ammonium acetate in 950ml of water and 50ml of acetonitrile. Store at room temperature, and discard after two months.

8.7 Dilution Mix - Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid

Add ammonium hydroxide (3.3ml of 30%), reagent water (1.7ml) and acetic acid (0.625ml) to methanol (92ml). Store at room temperature, replace after 1 month.

8.8 Nitrogen – various grades

8.9 Per fluorinated Alkyl Substances stock standards – Traceable to Certificate of Analysis.

8.10 Qualitative Identification Standards- br-PFNA and T-PFOA from Wellington Laboratories.

8.11 Bile salt interference check standard- Taurodeoxycholic Acid (TDCA) or Sodium taurodeoxychloate hydrate – (Sigma Aldrich 580221-5GM, or equivalent). Prepare solution at a concentration of 1 µg/mL in the Acetonitrile.

8.12 Mass labeled – Non-Extracted Internal Standards

$^{13}C_3$-PFBA	$^{13}C_4$-PFOA	$^{13}C_2$-PFDA	$^{13}C_4$-PFOS
$^{13}C_2$-PFHxA	$^{13}C_5$-PFNA	$^{18}O_2$-PFHxS	

8.13 Mass labeled – Extracted Internal Standards

$^{13}C_4$-PFBA	$^{13}C_9$-PFNA	$^{13}C_3$-PFBS	$^{13}C_2$-8:2 FTS	D_5-NEtFOSAA
$^{13}C_5$-PFPeA	$^{13}C_6$-PFDA	$^{13}C_3$-PFHxS	$^{13}C_8$-PFOSA	D_7-NMeFOSE
$^{13}C_5$-PFHxA	$^{13}C_7$-PFUnA	$^{13}C_8$-PFOS	D_3-NMeFOSA	D_9-NEtFOSE
$^{13}C_4$-PFHpA	$^{13}C_2$-PFDoA	$^{13}C_2$-4:2 FTS	D_5-NEtFOSA	$^{13}C_3$-HFPO-DA
$^{13}C_8$-PFOA	$^{13}C_2$-PFTeDA	$^{13}C_2$-6:2 FTS	D_3-NMeFOSAA	

9.0 INTERFERENCES

9.1 Data from all blanks, samples, and spikes must be evaluated for interferences. Method interferences may be caused by contaminants in solvents, reagents, or glassware. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) or Teflon products, HPLC solvent lines, methanol, aluminum foil, SPE transfer lines, bottle caps, etc. All materials must be demonstrated to be free from interferences.

9.2 Contact with glass containers, pipettes, or syringes should be minimized since the Perfuorinated compounds can potentially adsorb to glass surfaces.

9.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample. Humic and/or fulvic material can be co-

extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of the humic content of the sample. High levels of iron have been shown to reduce the d5-EtFOSAA recoveries.

9.4 When establishing the chromatographic conditions, it is important to consider the potential interference of bile salts during analyses of tissue samples. A standard containing TDCA should be injected to ensure that TDCA does not coelute with any of the target analytes, EIS, or NIS standards. Analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and PFOS.

9.5 SPE cartridges can be a source of interferences. The analysis of field and method blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices must be tested to ensure that contamination does not preclude analyte identification and quantitation.

9.6 Water and containers used for equipment blanks or field blanks must be tested prior to use. For smaller sampling events DI water will be provided in the same type of bottle used for sample collection. For larger sampling events four-liter HDPE containers should be used. Containers should be filled with DI water and allowed to sit for several hours before testing. If the bottles are from the same lot and filled with DI on the same day, then one analysis per 10 containers should suffice. The DI water and container blanks must be free of any analytes of interest or interferences below the MDL.

9.7 A field blank should be collected with each set of samples. Each field blank consists of 4 bottles. Two bottles are filled with DI water at the lab and the other two bottles are empty. At the sampling site the sampler should open then two empty bottles and transfer the DI water from the full bottles into them. Cap the bottles, label as field blanks, and return them to the laboratory along with the samples for analysis.

10.0 PROCEDURE

10.1 Standards Preparation. **Standards and sample extracts must be brought to room temperature and vortexed prior to aliquoting into an instrument vial.**

10.1.1 Standards are prepared from commercially available certified neat or reference standards. All standards must be logged in the HPLC Standards Logbook. All standards shall be traceable to their original source. The standards must be stored at ≤ 6°C, or as recommended by the manufacturer. Calibration levels, spike and isotope dilution standard concentrations, preparation information, and vendor part numbers can be found in the LCMS STD Summary in the Active SOP directory. A summary of the calibration concentrations can be found in Table 3.

10.2 Stock Standard Solutions

10.2.1 Stock standards are available from some commercial vendors. All vendors must supply a “Certificate of Analysis” with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor’s expiration date. Once opened, the hold time is reduced to one year or the vendor’s expiration

date (whichever is shorter).

10.3 Intermediate Standard Solutions

10.3.1 Intermediate standards are prepared by quantitative dilution of the stock standard with methanol. The hold time for intermediate standards is six months or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparisons to other standards indicate analyte degradation or concentration changes. Intermediate standards should be prepared using the dilution mix and stored in polyethylene vials.

10.4 Calibration Standards

10.4.1 Calibration standards for Perfluorinated analytes are prepared at a minimum of six concentration levels through quantitative dilutions of the intermediate standard. Calibration standards are prepared in methanol. The low standard is at a concentration at or below the RL and the remaining standards define the working range of the detector. Calibration standards should be prepared using the dilution mix and be stored in polyethylene vials. See Table 3 for levels.

10.4.2 Calibration standards concentrations for the sulfonates may need to be corrected for the molecular weight of the cation in the salt. Check the vendor's Certificate of Analysis to see if their nominal concentration is based on the acid or salt.

$$Mass_{acid} = Mass_{salt} \mathbf{X} MW_{acid}/MW_{salt}$$

MW_{acid} = Molecular weight of PFAA
MW_{salt} = Molecular weight of the salt

NOTE: Per fluorinated analytes may exist in branched and/or linear form. If a branched form is commercially available, then the calibration standards must contain the branched and linear form. The Quantitative standards must contain PFHxS, PFOS, PFOSA, NMeFOSAA, NEtFOSAA NMeFOSA, NEtFOSA, NMeFOSE, NEtFOSE, PFOA and PFNA which are currently available in mixes of branched and linear isomers.

10.4.3 Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

10.5 HPLC/MS/MS Conditions

10.5.1 HPLC Conditions

10.5.1.1 6-10ul autosampler injection
10.5.1.2 Gradient Program
10.5.1.3 Eluent A – Acetonitrile
10.5.1.4 Eluent B – 2mM ammonium acetate in 95:5 water: acetonitrile
10.5.1.5 Column temperature – 50.0 °C

Time (min)	A (%)	B (%)	Flow (mL/min)
0.20 min	10.0 %	90.0 %	0.350 mL/min
4.00 min	30.0 %	70.0 %	0.350 mL/min
7.00 min	55.0 %	45.0 %	0.350 mL/min
9.00 min	75.0 %	25.0 %	0.350 mL/min
10.00 min	95.0 %	5.0 %	0.400 mL/min
10.30 min	95.0 %	5.0 %	0.400 mL/min
10.40 min	2.0 %	98.0 %	0.400 mL/min
11.80 min	2.0 %	98.0 %	0.400 mL/min
13.00 min	2.0 %	98.0 %	0.350 mL/min

10.5.2 MS/MS Conditions

Parameter	Value	Parameter	Value
Gas Temp C	250	Sheath Gas Flow (l/min)	10
Gas Flow (l/min)	10	Capillary (V)	3500
Nebulizer (psi)	50	V Charging	500
Sheath Gas Heater	300	Ionization Mode	Neg ESI
Collision Cell Gas (psi)	40	Collision Cell Gas	UHP N2

Fragmentation voltages and collisions energies are optimized for each analyte and are stored in the instrument method. Precursor ions and transition masses are listed in Table 2.

LC/MS/MS conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

10.6 Sample Preparation

10.6.1 Low Level Aqueous Samples

10.6.1.1 A 500ml aliquot of sample (entire bottle) is extracted utilizing a solid phase extraction cartridge. The cartridge is eluted with basic methanol. The extract is carbon cleaned, filtered and the final volume is adjusted to 5.0ml, and then transferred to a centrifuge tube for storage. Refer to SOP EOP041.

10.6.2 Solid Samples

10.6.2.1 A 5-gram aliquot sample is extracted with basic methanol utilizing vortex mixer and a shaker table. The extract is carbon cleaned, SPE cleaned, filtered and the final volume is adjusted to 5.0ml, and then transferred to a centrifuge tube for storage. Refer to SOP EOP040.

10.7 HPLC/MS/MS Analysis

Instrument calibration consists of four major sections:

Mass Tuning and Calibration Transition Window Selection Initial Calibration Procedures Continuing Calibration Verification

10.7.1 Mass Calibration and Transition Window Selection

The instrument must have a valid mass calibration prior to any sample analysis. The mass calibration must be updated as needed. (i.e. QC failures, ion masses showing large deviations from known masses, or after major instrument maintenance is performed). It is recommended that the mass calibration be verified weekly through the analysis of a Check Tune. The Agilent Check Tune Masses range from 112.99 to 2233.91 amu for MS1 and 69.00 to 2233.91 for MS2.

The Check Tune Report may show both Positive and Negative ESI Results. Only the Negative results need to be evaluated. Unit resolution is demonstrated when the value of the peak width at half-height is within 0.5 ± 0.1 amu of the true value.

MS1 (UNIT)	MS2 (UNIT)
	69.00
112.99	112.99
302.00	302.00
601.98	601.98
1033.99	1033.99
1633.95	1633.95
2233.91	2233.91

Since masses greater than 1033.99 amu are not used for this method, the 1633.95 and 2233.91 amu masses must be present but do not need to be within 0.1 amu of the true value.

The Branched/Linear RT Check and mid-point calibration standard are used to check the analyte retention times. These retention times are used to update the transition windows. The windows must be wide enough to ensure that the branched and linear isomer PFAS analytes are completely within the transition window. The branched isomers will elute just prior to the linear isomer. If they are partially cut off, adjust the retention time of the linear isomer or the width of the transition window. Use a similar size window for the other analytes that do not have a branched standard. Later eluting peaks are broader and require a slightly wider transition windows because of peak broadening.

10.7.2 Initial Calibration Procedures

Before samples can be run, the LC/MS/MS system must be calibrated. The calibration must be performed every time after taking an action that changes the chromatographic conditions or when either ICV/CCV or Instrument Sensitivity check don’t meet acceptance criteria. The signal to noise ratio must be >= 3:1 for analytes with both a quant and confirmation ion and >= 10:1 for analytes with no confirmation ion.

10.7.2.1 Isotope Dilution Standard (Extracted Internal Standard) Calibration

A minimum 6-point calibration curve is created for the native PFAS compounds using an Isotope Dilution or Extracted Internal Standard technique. SGS - Dayton routinely performs an 8-point calibration to maximize the calibration range and to allow for quadratic fits. See Table 3.

The calibration standards for PFHxS, PFOS, PFOA, PFNA, PFOSA, NMeFOSAA, NEtFOSAA NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE must consist of both branched and linear isomers. The branched isomer elutes just prior to the linear isomer. These 9 PFAS are currently being reported as the sum of the branched and linear isomers so both the branched and linear isomers in the calibration standards must be integrated.

Response factors (RF) for each analyte at each calibration level are determined as follows:

$RF = (A_{analyte}\ C_{ids})/(A_{ids} \mathbf{X}\ C_{analyte})\ \ A_{analyte}$

= area of the analyte
A_{ids} = area of the isotope dilution standard $C_{analyte}$
= concentration of the analyte
C_{ids} = concentration of the isotope dilution standard.

The mean RF and standard deviation of the RF are determined for each analyte and EIS. The percent relative standard deviation (%RSD) of the response factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of RF X 100) / Mean RF

If the %RSD $\leq$ 20%, linearity through the origin can be assumed and the mean RF can be used to quantitate target analytes in the samples.

Alternatively, a weighted linear regression or non-linear regression may be used. If used, the regression must be weighted inversely proportional to concentration and must not be forced through zero. The correlation coefficient (r value) must be $\geq$ 0.995 or ±0.99 (for r^2 value) for each compound to be acceptable. If a linear or non-linear regression is used, then the Relative Standard Error (%RSE) must be calculated.

Calculation of Relative Standard Error (%RSE)

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i}\right]^2 / (n-p)}$$

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at calibration level i, in mass or *concentration units.*

p = Number of terms in the fitting equation. (average = 1, linear = 2, quadratic = 3)

n = Number of calibration points.

If Relative Standard Error (%RSE) ≤ 20%, then the curve can be used to quantitate target analytes in the samples.

NOTE: If any EIS was removed from a specific calibration point to meet the acceptance criteria for the initial calibration, then corresponding native compound in that calibration point must be also removed.

10.7.2.2 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV must be prepared from a second source at a mid-range concentration.

NOTE: Second source standards may consist of linear isomers only.

The %D for the compound of interest must be ≤ ±30% (70-130% of True Value). If the ICV does not meet criteria, a fresh standard must be prepared. If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, make fresh calibration standards. Recalibrate the instrument.

NOTE: Analyze the branched/linear standard to identify the branched isomers. This is a qualitative standard only. Currently it should contain branched isomers of PFOA and PFNA. This standard is loaded into LIMS as an ICV.

10.7.2.3 Bile Salt Interference Check and Branched/Linear Retention Time Check.

The separation between Taurodeoxycholic Acid (TDCA) and PFOS must be verified with each ICAL.

For QSM 5.4 the separation between Taurodeoxycholic Acid (TDCA) and PFOS must be verified daily.

Inject a mid-level PFAS standard that has been fortified with 1 ug/ml TDCA. The standard may also contain Taurochenodeoxycholic Acid (TCDCA) and Tauroursodeoxycholic Acid (TUDCA) as well.

TDCA must be separated by at least 1 minute from all branched and linear isomers of PFOS.

10.7.2.4 Branched/Linear RT Check. Analyze

The branched/linear RT standard to identify the branched isomers. This is a qualitative standard only. Currently, it should contain branched isomers of PFOA and PFNA. This standard is loaded into LIMS as an RT Check.

10.7.2.5 Highest Standard and Instrument Blank

Analyze an instrument blank (IBLK) immediately following the highest standard analyzed. The highest standard analyzed may be analyzed as part of the calibration curve or following the calibration curve. The highest standard may be at or above the concentration of highest level of calibration. It cannot be used to extend the calibration range.

The instrument blank must be analyzed immediately following the highest standard. The instrument blank must be free of any analytes of interest or interferences at or below the MDL to be acceptable.

If the acceptance criteria is not met, the concentration of the standard should be lowered and another blank analyzed.

The highest standard and instrument blank pair are used only to document the highest concentration at which carryover does not occur. If a sample concentration exceeds this range and the sample(s) following have reportable detections for that analyte, then they must be reanalyzed.

10.7.2.6 Retention Time Windows

The retention time of each analyte and extracted internal standard must fall within **0.4 minutes** of the predicted retention times from the daily calibration verification or from the midpoint standard of the ICAL (on days when an ICAL is performed).

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and extracted internal standard from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use

the retention time of the mid-point standard of the initial calibration.

Initial peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (extracted internal standards) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations. HPLC retention times tend to shift more than GC retention times.

The retention time of the target analyte must fall within **0.1 minutes** of the associated isotope dilution standard (for analytes that have an exact isotopic counterpart).

10.7.2.7 Ion Ratios and Signal to Noise

A minimum of two transition ions are monitored for each target analyte except for those analytes in Table 2 which only have a single transition ion.

The ratio of the primary and secondary transition masses should be updated from the initial calibration. They may be updated from the midpoint standard or from an average of all levels. Additionally, the ion ratio may be updated from the opening daily CCV.

Isotope Ratio criteria is still being developed for EPA method 1633. The MassHunter software calculates the ratio as the response of the primary transition mass divided by the response of the secondary transition mass times 100. It is set to flag the analyte if the ratio of these ions is not within ± 50% of the expected, (e.g., if the ion ratio is expected to be 50% in the standard, the ion ratio in the corresponding sample must be between 25 and 75%).

Primary and secondary transition masses must maximize within ± 2 seconds.

The signal to noise ratio for the primary transition mass must be at least 3 times that of the background and the secondary transition mass must be at least 3 times that of the background.

10.7.3 Daily Calibration and Carryover Verifications

10.7.3.1 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for the Perfluorinated compounds are prepared at low and mid-range concentration. CCV standards are prepared from the same stock as the initial calibration standards.

A low level CCV (Instrument Sensitivity check) must be analyzed

at the beginning of each analytical sequence (prior to sample analysis) and at least once every 24 hours during the sequence to ensure accuracy at the LOQ.

The CCV must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally, the mid-point CCV must be analyzed after every 10 samples.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| must be ≤ 30% for the target analytes and EIS in each CCV.

If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet

criteria, the system must be recalibrated. If the second standard meets criteria, then a third standard must be analyzed. If the third standard also meets criteria, then the system is considered in control and results may be reported.

If the |%D| is outside the control limits, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Qualifying the data should only be done if the sample cannot be reanalyzed. Under certain circumstances, the data may be reported,
i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed, or the data must be qualified.

10.7.3.2 Carryover Verification

A high standard and an instrument blank (IBLK) must be analyzed each day prior to the analysis of samples. The high standard may be at or above the concentration of highest level of the calibration.

The instrument blank must be analyzed immediately following the high standard. The instrument blank must be free of any analytes of interest or interferences at or below the MDL to be acceptable.

If the acceptance criteria are not met, the concentration of the standard should be lowered, and another blank analyzed.

The highest standard and instrument blank pair are used only to

document the highest concentration at which carryover does not occur. If sample concentrations exceed this range and the sample(s) following exceed this acceptance criteria (>1/2 LOQ), they must be reanalyzed.

10.7.3.3 Continuing Calibration Blank (CCB)

An additional blank must be analyzed after each CCV to ensure no carryover from the standard. The instrument blank must be free of any analytes of interest or interferences at ½ the required LOQ to be acceptable. The CCB is loaded into LIMS as "ICCB".
If the acceptance criteria are not met, the system should be checked. Any samples bracketed by the failing CCB must be reanalyzed.

Review the data to see if there was a high sample prior to the CCV/CCB pair that may have contaminated the system? If so, clean the system and run additional blanks to see if the system is in control.

10.7.3.4 Bile Salt Interference Check.

Inject a mid-level PFAS standard that has been fortified with 1 ug/ml TDCA. The standard may also contain Taurochenodeoxycholic Acid (TCDCA) and Tauroursodeoxycholic Acid (TUDCA) as well.

TDCA must be separated by at least 1 min relative to all of the branched and linear isomers of PFOS.

10.7.3.5 Branched.Linear RT Check

Analyze the branched/linear RT standard daily to identify the branched isomers. This is a qualitative standard only. Currently, it should contain branched isomers of PFOA and PFNA. This standard is loaded into LIMS as an RT Check

10.7.4 Sample Extract Analysis

10.7.4.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Initial Calibration Standards
ICV
Carryover Check Standard
Instrument Blank (IBLK)
Bile Salt Interference Check
Branched/Linear RT
CCV Standards:
Low-Level (LOQ)- Instrument Sensitivity check
Mid-Level

QC Extracts
Sample Extracts
Bracketing CCV
Bracketing CCB

10.7.4.2 Six to ten microliters (same amount as standards) of extract is injected into the HPLC by the autosampler. The data system then records the resultant peak responses and retention times.

10.7.4.3 Tentative identification of an analyte occurs when the peak from the sample extract falls within the retention time window of the target compound.

10.7.4.4 Positive identification is confirmed by comparing the ion ratio in the sample to the ion ratio of the standards. For the linear isomer, the primary and secondary transition masses must both be present. For the branched isomers the primary and secondary transition masses should both be present. In rare circumstances a particular branched peak may only exhibit the primary transition ion. These should be omitted from the quantitation.

The MassHunter software is set to flag the analyte if the ratio of these ions is not within ± 30% of the expected, (e.g., if the ion ratio is expected to be 50% in the standard, the ion ratio in the corresponding sample must be between 20 and 80%).

The signal to noise ratio for the primary transition mass must be at least 3 times that of the background and the secondary transition mass must be at least 3 times that of the background.

10.7.4.5 Some of the PFASs may have multiple chromatographic peaks due to the presence of linear and branched isomers. This is prevalent in PFHxS and PFOS. The areas of all the linear and branched isomers peaks must be included and the concentrations reported as a total for each of these analytes.

NOTE: The branched isomers for PFOA and PFNA must be included in the quantitation even if the calibration is based on just the linear isomer.

10.7.4.6 If the compound identification does not confirm, then the result should be reported as ND.

10.7.4.7 If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

Dilutions for this method are performed differently depending on the concentration of the target analytes in the extract. For dilutions in the 2x to 10x range, the extract is diluted with the

dilution mix. No additional EIS nor NIS are added. NIS concentrations in the diluted samples are normalized by multiplying calculated concentration by dilution factor.

If the responses for each EIS in the diluted extract meet the S/N requirements in Section 10.7.2 and retention time requirements in Section 10.7.2.6, and the EIS recoveries from the analysis of the diluted extract are greater than 5%, then the compounds associated with those EISs may be quantified using isotope dilution.

Use the EIS recoveries from the original analysis to select the dilution factor, with the objective of keeping the EIS recoveries in the dilution above that 5% lower limit (i.e., if the EIS recovery of the affected analyte in the undiluted analysis is 50%, then the sample cannot be diluted more than 10:1; if the if the EIS recovery of the affected analyte in the undiluted analysis is 30%, then the sample cannot be diluted more than 6:1).

For dilutions greater than 10-fold, a smaller aliquot should be extracted for soil samples. The estimated analyte concentration from below can be used to determine the best aliquot size. The method requires the collection of a smaller sample size for aqueous samples in such cases (i.e., 125 mL). If aliquoting of an aqueous sample is necessary, the client must be notified prior to subsampling.

If no additional sample is available, then additional EIS and NIS are added, and the sample re-analyzed. The theoretical concentration of the isotope dilution standards in the extract will need to be entered into MassHunter so that the software can correctly calculate the native analyte concentration. This result is estimated based on an internal standard approach. The results should be footnoted as such.

10.7.4.8 If peak identification is prevented by the presence of interferences, further cleanup may be required, or the extract must be diluted so that the interference does not mask any analytes.

10.8 Maintenance and Trouble Shooting

10.8.1 All instrument maintenance must be documented in the appropriate “Instrument Repair and Maintenance” log. The log will include such items as problem, action taken, correction verification, date, and analyst.

10.8.2 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.

10.8.3 PC and software changes must be documented in the “Instrument Repair and Maintenance” log. Software changes may require additional validation.

11.0 METHOD PERFORMANCE

11.1 Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), low-level blank spikes (LLBS), matrix spikes (MS), matrix spike duplicates (MSD) and sample duplicates (DUP). The MB, BS, LLBS are used to monitor overall method performance, while the MS and MSD or DUP are used to evaluate the method performance and reproducibility in a specific sample matrix.

11.2 The QC sample recoveries for water samples are compared to the required QC acceptance limits in table 5 and 6. The same limits must be used for soil samples until in-house limits can be developed by the laboratory.

12.0 QC REQUIREMENTS

Accuracy and matrix bias are monitored by the use of isotope dilution standards and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), matrix spike duplicate (MSD) or sample duplicate (DUP). All control limits are updated annually and are listed in the LIMS.

12.1 Initial Demonstration of Capability.
To establish the ability to generate acceptable precision and recovery, the laboratory must perform the following: four blank spikes (BS) and metho blank (MB) must be prepared and analyzed for each matrix type to be tested. Using the results, the average recovery and RSD (Relative Standard Deviation) are calculated. The average percent recovery and RSD must meet the requirements in the Table 5.

12.2 Non-Extracted Internal Standards (NIS)

12.2.1 The analytes listed in section 5.10 are used as the Non-Extracted Internals Standards for this method. The response of the NIS in all subsequent runs must be 50-200% of the average response from the initial calibration (see Table 5).

12.2.2 If the NIS responses are not within limits, the following are required.

12.2.2.1 Check to be sure that there are no errors in calculations, integrations, or internal standards solutions. If errors are found, recalculate the data accordingly.

12.2.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem, and reanalyze the sample. If no problem is found, prepare a second aliquot of extract, and reanalyze the sample.

12.2.2.3 If upon reanalysis, the responses are still not within limits reanalyze the sample at a dilution.

12.2.2.4 If upon analysis of the dilution the responses are within limits, then the sample or select analytes may need to

be reported from the dilution or qualified.

12.3 Extracted Internal Standard (EIS)

12.3.1 The analytes listed in section 5.11 are used as the Extracted Internal Standards for this method.

A known amount of isotope dilution standard is added to each sample including the QC set prior to extraction. The recovery (corrected for dilution) for each isotope dilution standard must follow the limits in Table 6 for water and soil matrices.

The % recovery is calculated from the calculated concentrations.

% Recovery = (Sample Amount / Amount Spiked) X 100

Only those isotope dilution standards that directly link to the native analytes being reported need to pass. For example, 13C4-PFBA only needs to pass if PFBA is being reported.

12.3.2 If any isotope dilution standard response/recovery is not within the established control limits, the following are required.

12.3.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, isotope dilution standard solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re- inject the extract to verify.

12.3.2.2 Check instrument performance. It may be necessary to re-vial and re- inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

12.3.2.3 Check for instrument suppression or enhancement by reanalyzing the sample at a dilution.

12.3.2.4 If no problem is found re-extract and reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary. If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

12.3.2.5 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference . Isotope dilution standards from both sets of analysis must be reported on the final report.

12.4 Method Blank

12.4.1 The method blank is either HPLC water or cleaned sand (depending upon

sample matrix). The method blank is then taken through all procedures along with the other samples to determine any contamination from reagents, glassware, or high- level samples. The method blank must be free of any analytes of interest or interferences at ½ the required LOQ to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier.

12.4.2 If the MB is contaminated but the samples are non-detect, then the source of contamination must be investigated and documented. The samples may need to be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

12.4.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination must be investigated and documented. The samples results may be reported with the appropriate “B” or “V” qualifier. This must be approved by the department supervisor.

12.4.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination must be investigated and documented. The samples must be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

12.5 Blank Spike

12.5.1 The blank spike is either HPLC water or cleaned sand (depending upon sample matrix) to which the spike standard has been added. The blank spike is then taken through all procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable (see Table 5). As additional analytes are added to this method, the recoveries will need to be carefully evaluated.

12.5.2 If the blank spike recoveries are not within the established control limits, the following are required.

12.5.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

12.5.2.2 Check instrument performance. It may be necessary to re-vial and re- inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

12.5.2.3 If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable.

12.5.2.4 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

12.5.2.5 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

12.6 Low-Level Blank Spike

12.6.1 The low-level blank spike is either HPLC water or cleaned sand (depending upon sample matrix) to which the spike standard has been added at no more than 2 times the LLOQ. The low-level blank spike is then taken through all procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable (see table 5). As additional analytes are added to this method, the recoveries will need to be carefully evaluated.

12.6.2 If the low-level blank spike recoveries are not within the established control limits, the following are required. Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

12.6.2.1 Check instrument performance. It may be necessary to re-vial and re- inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

12.6.2.2 If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable.

12.6.2.3 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as

estimated.

12.6.2.4 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

12.7 Matrix Spike and Matrix Spike Duplicate/Matrix Duplicate

12.7.1 Matrix spike and spike duplicates are second samples to which the spike standard has been added. The matrix spike and spike duplicate or matrix duplicate are then taken through all procedures along with the other samples to monitor the precision and accuracy of the procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable (see table 5).

12.7.2 If the matrix spike recoveries are not within the established control limits, the following are required.

12.7.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

12.7.2.2 Check instrument performance. It may be necessary to re-vial and re- inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

12.7.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extraction but are indications of the sample matrix effects.

12.7.3 Precision

Matrix spike and spike duplicate or sample and matrix duplicate recoveries for each analyte OR sample result and duplicate result are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each Perfluorinated compound must be less than 30%. If the RPDs fall outside of the established control limits, the MS/MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and

determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

The RPD for each Per fluorinated compound must be less than 30%. If the RPDs fall outside of the established control limits, the DUP should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

12.7.4 Accuracy assessment of the recovery of EIS and NIS compounds.

After the analysis of 30 samples of a given matrix type (water, solids, etc.), the lab must compute the recovery and the standard deviation of the percent recovery (SR) for the isotopically labeled compounds only. The assessment is expressed as a percent recovery interval from R - 2SR to R + 2SR for each matrix. The records must be maintained and assessed periodically(after every 10 new preparation batches).

13.0 CALCULATIONS

The concentration of each Perfluorinated compound in the original sample is calculated as follows:

Water (ug/l) = ($CONC_{inst}$) X (V_F / V_I) X DF

Soil (ug/kg) = [($CONC_{inst}$) X (V_F / W_I) X DF] / %solids

$CONC_{inst}$	=	Instrument concentration calculated from the initial calibration using mean CF or curve fit (ppb)
DF	=	Dilution Factor
V_F	=	Volume of final extract (ml)
V_I	=	Volume of sample extracted (ml)
W_I	=	Weight of sample extracted (g)
% solids	=	Dry weight determination in decimal form

14.0 DOCUMENTATION

14.1 The Analytical Logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

14.1.1 If samples require reanalysis, a brief explanation of the reason must be documented in this log.

14.1.2 Overwriting of data files is never allowed.

14.2 The Standard Preparation Logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

14.2.1 The SGS Lot Number must be cross-referenced on the standard vial.

14.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

14.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

14.5 Unused blocks of any form must be X'ed or Z'ed out by the analyst before submitting the data for review.

14.6 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information must also be verified during this review.

15.0 DATA REVIEW AND REPORTING

15.1 Initial and continuing calibration check. Verify that all calibration and continuing calibration criteria have been achieved. If the criteria had not been achieved, corrective action must be performed to bring the system in control before analyzing any samples.

15.1.1 If samples had been analyzed under non-compliant calibration criteria, all sample extracts must be re-analyzed once the system is brought into control.

15.2 Quality Control Data Review. Review all QC data. If QC criteria were not achieved, perform corrective action before proceeding with analysis.

15.2.1 In some situation, corrective action may demand that the entire sample batch be re-extracted and re-analyzed before processing data.

15.3 Chromatogram Review. The chromatogram of each sample is evaluated for target analytes.

15.3.1 Each sample may require the reporting of different target analytes. Review the login to assure that the correct target compounds are identified.

15.3.2 Manual integration of chromatographic peaks must be identified by the analysts. Upon review, the supervisor will initial and date the changes made to the report.

15.4 Transfer to LIMS. Following the initial screen review, transfer the processed data to the LIMS.

15.4.1 Compare the printed values to the original values to verify transfer accuracy.

15.4.2 If transfer errors occured, the errors must be corrected before the data is re-submitted.

15.4.3 The results in LIMS are reported in ug/L for water samples and in ug/Kg on a dry-weight basis for soil samples. All results in the samples and blanks are reported to the MDL as per the laboratory policy (refer to the SOP EQA035).

16.0 POLLUTION PREVENTION & WASTE MANAGEMENT

16.1 Wastewater and acetonitrile from the instrument are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

16.2 Sample Extracts are archived and stored for 30 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

17.0 REFERENCES

17.1 EPA Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, January 2024.

Table 1 : Target Analytes

PFAS Analyte	Acronym	CAS #
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTriA	72629-94-8
Perfluorotetradeconoic acid	PFTeA	376-06-7
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoDS	79780-39-5
4:2 Fluorotelomer sulfonate	4:2 FTS	757124-72-4
6:2 Fluorotelomer sulfonate	6:2 FTS	27619-97-2
8:2 Fluorotelomer sulfonate	8:2 FTS	39108-34-4
3:3 Fluorotelomer carboxylate	3:3 FTCA	356-02-5
5:3 Fluorotelomer carboxylate	5:3 FTCA	914637-49-3
7:3 Fluorotelomer carboxylate	7:3 FTCA	812-70-4
N-ethyl perfluorooctanesulfonamido acetic acid	EtFOSAA	2991-50-6
N-methyl perfluorooctanesulfonamido acetic acid	MeFOSAA	2355-31-9
Perfluorooctane sulfonamide	PFOSA	754-91-6
N-Ethyl perfluorooctane sulfonamide	EtFOSA	4151-50-2

N-Methyl perfluorooctane sulfonamide	MeFOSA	31506-32-8
N-Ethyl perfluorooctane sulfonamidoethanol	EtFOSE	1691-99-2
N-Methyl perfluorooctane sulfonamidoethanol	MeFOSE	24448-09-7
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6
11-chloroicosafluoro-3-oxaundecade-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	9Cl-PF3ONS	756426-58-1
4,8-dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6
Perfluoro(2-ethoxyethane) sulfonic acid	PFEESA	113507-82-7
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5

TABLE 2: Precursor and Primary Transition Masses

Analyte	Type	RT	Primary Transition	Secondary Transition	Reference Compound
13C3-PFBA	NIS	3.34	216.0 -> 172.0		
13C4-PFBA	EIS	3.34	216.8 -> 171.9		13C3-PFBA
PFBA	Target	3.34	212.8 -> 168.9		13C4-PFBA
PFMPA	Target	3.99	229.0 -> 84.9		13C5-PFPeA
3:3FTCA	Target	4.32	241.0 -> 177.0	241.0 -> 117.0	13C5-PFPeA
13C5-PFPeA	EIS	4.93	268.3 -> 223.0		13C2-PFHxA
PFPeA	Target	4.93	263.0 -> 219.0		13C5-PFPeA
PFMBA	Target	5.38	279.0 -> 85.1		13C5-PFPeA
13C2-4:2FTS	EIS	5.85	329.1 -> 80.9		18O2-PFHxS
4:2FTS	Target	5.85	327.1 -> 307.0	327.1 -> 80.9	13C2-4:2FTS
NFDHA	Target	6.08	295.0 -> 201.0	295.0 -> 84.9	13C5-PFHxA
13C3-PFBS	EIS	6.15	302.1 -> 79.9		18O2-PFHxS
PFBS	Target	6.15	298.7 -> 79.9	298.7 -> 98.8	13C3-PFBS
13C2-PFHxA	NIS	6.20	315.1 -> 270.0		
13C5-PFHxA	EIS	6.20	318.0 -> 273.0		13C2-PFHxA
PFHxA	Target	6.20	313.0 -> 269.0	313.0 -> 118.9	13C5-PFHxA
13C3-HFPO- DA	EIS	6.59	286.9 -> 168.9		13C2-PFHxA
HFPO-DA	Target	6.59	284.9 -> 168.9	284.9 -> 184.9	13C3-HFPO-DA
PFEESA	Target	6.71	314.8 -> 134.9	314.8 -> 82.9	13C5-PFHxA
5:3FTCA	Target	6.82	341.0 -> 237.1	341.0 -> 217.0	13C5-PFHxA
13C4-PFHpA	EIS	7.14	367.1 -> 322.0		13C2-PFHxA

PFHpA	Target	7.14	363.1 -> 319.0	363.1-> 169.0	13C4-PFHpA
PFPeS	Target	7.22	349.1 -> 79.9	349.1 -> 98.9	13C3-PFHxS
ADONA	Target	7.40	376.8 -> 250.9	376.8 -> 84.8	13C3-HFPO-DA
13C2-6:2FTS	EIS	7.56	429.1 -> 80.9		18O2-PFHxS
6:2FTS	Target	7.56	427.1 -> 407.0	427.1 -> 80.9	13C2-6:2FTS
13C4-PFOA	NIS	7.81	417.1 -> 172.0	417.1 -> 372.0	
13C8-PFOA	EIS	7.81	421.0 -> 376.0		13C4-PFOA
PFOA	Target	7.81	413.0 -> 369.0	413.0 -> 169.0	13C8-PFOA
PFHxS	Target	7.96	398.9 -> 79.9	398.9 -> 98.9	13C3-PFHxS
18O2-PFHxS	NIS	7.97	403.0 -> 83.9		
13C3-PFHxS	EIS	7.97	402.1 -> 79.9		18O2-PFHxS
7:3FTCA	Target	8.27	441.0 -> 316.9	441.0 -> 336.9	13C5-PFHxA
13C5-PFNA	NIS	8.40	468.0 -> 427.0		
13C9-PFNA	EIS	8.40	472.1 -> 427.0		13C5-PFNA
PFNA	Target	8.40	463.0 -> 419.0	463.0 -> 219.0	13C9-PFNA
PFHpS	Target	8.58	449.0 -> 79.9	449.0 -> 98.8	13C8-PFOS
13C2-8:2FTS	EIS	8.69	529.1 -> 80.9		18O2-PFHxS
8:2FTS	Target	8.70	527.1 -> 507.0	527.1 -> 80.8	13C2-8:2FTS
13C2-PFDA	NIS	8.95	515.1 -> 470.1		
13C6-PFDA	EIS	8.95	519.1 -> 474.1		13C2-PFDA
PFDA	Target	8.95	512.9 -> 469.0	512.9 -> 219.0	13C6-PFDA
d3-MeFOSAA	EIS	8.97	573.2 -> 419.0		13C4-PFOS
MeFOSAA	Target	8.97	570.1 -> 419.0	570.1 -> 483.0	d3-MeFOSAA
13C4-PFOS	NIS	9.14	503.8 -> 79.9		
13C8-PFOS	EIS	9.13	507.1 -> 79.9		13C4-PFOS
PFOS	Target	9.14	498.9 -> 79.9	498.9 -> 98.8	13C8-PFOS
d5-EtFOSAA	EIS	9.19	589.2 -> 419.0		13C4-PFOS
EtFOSAA	Target	9.20	584.2 -> 419.1	584.2 -> 526.0	d5-EtFOSAA
13C7-	EIS	9.44	570.0 -> 525.1		13C2-PFDA
PFUnDA	Target	9.44	563.1 -> 519.0	563.1 -> 269.1	13C7-PFUnDA
9Cl-PF3ONS	Target	9.49	530.8 -> 351.0	532.8 -> 353.0	13C3-HFPO- DA
PFNS	Target	9.63	548.8 -> 79.9	548.8 -> 98.8	13C8-PFOS
13C2-	EIS	9.87	615.1 -> 570.0		13C2-PFDA
PFDoDA	Target	9.87	613.1 -> 569.0	613.1 -> 319.0	13C2-PFDoDA
PFDS	Target	10.05	599.0 -> 79.9	599.0 -> 98.8	13C8-PFOS
13C8-FOSA	EIS	10.23	506.1 -> 77.8		13C4-PFOS
FOSA	Target	10.23	498.1 -> 77.9	498.1 -> 478.0	13C8-FOSA
PFTrDA	Target	10.26	663.0 -> 619.0	663.0 -> 168.9	13C2-PFDoDA
11Cl-	Target	10.32	630.9 -> 451.0	632.9 -> 453.0	13C3-HFPO- DA
13C2-PFTeDA	EIS	10.60	715.1 -> 670.0		13C2-PFDA
PFTeDA	Target	10.60	713.1 -> 669.0	713.1 -> 168.9	13C2-PFTeDA

PFDoDS	Target	10.75	699.1 -> 79.9	699.1 -> 98.8	13C8-PFOS
d7-MeFOSE	EIS	11.21	623.1 -> 58.9		13C4-PFOS
MeFOSE	Target	11.22	616.1 -> 58.9		d7-MeFOSE
d3-MeFOSA	EIS	11.30	515.0 -> 219.0		13C4-PFOS
MeFOSA	Target	11.30	512.0 -> 219.0	512.0 -> 169.0	d3-MeFOSA
d9-EtFOSE	EIS	11.45	639.1 -> 58.9		13C4-PFOS
EtFOSE	Target	11.46	630.0 -> 58.9		d9-EtFOSE
d5-EtFOSA	EIS	11.53	531.1 -> 219.0		13C4-PFOS
EtFOSA	Target	11.53	526.0 -> 219.0	526.0 -> 169.0	d5-EtFOSA

TABLE 3: Standard Levels (Targets), ng/ml

Compound	CS1	CS2	CS3	CS4 (CV1)	CS5	CS6	CS7	CS8
Perfluoroalkyl carboxylic acids								
PFBA	0.8	1.6	5.0	10	20	50	100	250
PFPeA	0.4	0.8	2.5	5	10	25	50	125
PFHxA	0.2	0.4	1.25	2.5	5.0	12.5	25	62.5
PFHpA	0.2	0.4	1.25	2.5	5.0	12.5	25	62.5
PFOA	0.2	0.4	1.25	2.5	5.0	12.5	25	62.5
PFNA	0.2	0.4	1.25	2.5	5.0	12.5	25	62.5
PFDA	0.2	0.4	1.25	2.5	5.0	12.5	25	62.5
PFUnA	0.2	0.4	1.25	2.5	5.0	12.5	25	62.5
PFDoA	0.2	0.4	1.25	2.5	5.0	12.5	25	62.5
PFTrDA	0.2	0.4	1.25	2.5	5.0	12.5	25	62.5
PFTeDA	0.2	0.4	1.25	2.5	5.0	12.5	25	62.5
Perfluoroalkyl sulfonic acids								
PFBS	0.177	0.354	1.109	2.218	4.435	11.087	22.17	55.438
PFPeS	0.188	0.376	1.176	2.353	4.705	11.762	23.52	58.813
PFHxS	0.183	0.366	1.143	2.285	4.570	11.425	22.85	57.125
PFHpS	0.191	0.382	1.191	2.383	4.765	11.912	23.82	59.563
PFOS	0.186	0.372	1.160	2.320	4.640	11.600	23.20	58.000
PFNS	0.192	0.384	1.203	2.405	4.810	12.025	24.05	60.125
PFDS	0.193	0.386	1.206	2.413	4.825	12.062	24.12	60.313
PFDoS	0.194	0.388	1.213	2.425	4.850	12.125	24.25	60.625
Fluorotelomer sulfonic acids								
4:2FTS	0.750	1.500	4.688	9.375	18.75	46.87	93.75	234.37
6:2FTS	0.760	1.520	4.750	9.500	19.00	47.50	95.00	237.50
8:2FTS	0.768	1.536	4.800	9.600	19.20	48.00	96.00	240.00
Perfluorooctane sulfonamides								
PFOSA	0.4	0.8	2.5	5	10	25	50	125
NMeFOSA	0.4	0.8	2.5	5	10	25	50	125
NEtFOSA	0.4	0.8	2.5	5	10	25	50	125
Perfluorooctane								
NMeFOSAA	0.2	0.4	1.25	2.5	5.0	12.5	25	62.5
NEtFOSAA	0.2	0.4	1.25	2.5	5.0	12.5	25	62.5
Perfluorooctane sulfonamide								
NMeFOSE	1	2.0	6.25	12.5	25	62.5	125	312.5
NEtFOSE	1	2.0	6.25	12.5	25	62.5	125	312.5
Per- and polyfluoroether								

HFPO-DA	0.8	1.6	5.0	10	20	50	100	250
ADONA	0.756	1.512	4.725	9.45	18.9	47.25	94.5	236.25
PFMPA	0.4	0.8	2.5	5.0	10	25	50	125
PFMBA	0.4	0.8	2.5	5.0	10	25	50	125
NFDHA	0.4	0.8	2.5	5.0	10	25	50	125
Ether sulfonic acids								
9Cl-PF3ONS	0.748	1.496	4.675	9.35	18.7	46.75	93.5	233.75
11Cl-PF3OUdS	0.756	1.512	4.725	9.45	18.9	47.25	94.5	236.25
PFEESA	0.356	0.712	2.225	4.45	8.90	22.25	44.5	111.25
Fluorotelomer carboxylic acids								
3:3FTCA	0.9984	1.997	6.24	12.48	25.0	62.4	124.8	312.0
5:3FTCA	4.992	9.984	31.20	62.4	124.8	312.0	624.0	1560
7:3FTCA	4.992	9.984	31.20	62.4	124.8	312.0	624.0	1560
Extracted Internal Standard (EIS)								
13C4-PFBA	10	10	10	10	10	10	10	10
13C5-PFPeA	5	5	5	5	5	5	5	5
13C5-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C4-PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C8-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C9-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C6-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C7-PFUnA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFTeDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C3-PFBS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C3-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C8-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C2-4:2 FTS	5	5	5	5	5	5	5	5
13C2-6:2 FTS	5	5	5	5	5	5	5	5
13C2-8:2 FTS	5	5	5	5	5	5	5	5
13C8-PFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D3-NMeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D5-NEtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
D3-NMeFOSAA	5	5	5	5	5	5	5	5
D5-NEtFOSAA	5	5	5	5	5	5	5	5
D7-NMeFOSE	25	25	25	25	25	25	25	25
D9-NEtFOSE	25	25	25	25	25	25	25	25
13C3-HFPO-DA	10	10	10	10	10	10	10	10
Non-extracted Internal Standard								
13C3-PFBA	5	5	5	5	5	5	5	5
13C2-PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C4-PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C5-PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
13C2-PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
18O2-PFHxS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
13C4-PFOS	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5

TABLE 4 : Method Names vs Lab Names

METHOD		LABORATORY		
ANALYTE NAME	ACRONYM	LIMS REPORT NAME	RAW DATA NAME	RAW DATA EIS as ISTD
Perfluorobutanoic acid	PFBA	Perfluorobutanoic acid	PFBA	
Perfluoropentanoic acid	PFPeA	Perfluoropentanoic acid	PFPeA	
Perfluorohexanoic acid	PFHxA	Perfluorohexanoic acid	PFHxA	
Perfluoroheptanoic acid	PFHpA	Perfluoroheptanoic acid	PFHpA	
Perfluorooctanoic acid	PFOA	Perfluorooctanoic acid	PFOA	
Perfluorononanoic acid	PFNA	Perfluorononanoic acid	PFNA	
Perfluorodecanoic acid	PFDA	Perfluorodecanoic acid	PFDA	
Perfluoroundecanoic acid	PFUnA	Perfluoroundecanoic acid	PFUnDA	
Perfluorododecanoic acid	PFDoA	Perfluorododecanoic acid	PFDoDA	
Perfluorotridecanoic acid	PFTrDA	Perfluorotridecanoic acid	PFTrDA	
Perfluorotetradecanoic acid	PFTeDA	Perfluorotetradecanoic acid	PFTeDA	
Perfluorobutanesulfonic acid	PFBS	Perfluorobutanesulfonic acid	PFBS	
Perfluoropentanesulfonic acid	PFPeS	Perfluoropentanesulfonic acid	PFPeS	
Perfluorohexanesulfonic acid	PFHxS	Perfluorohexanesulfonic acid	PFHxS	
Perfluoroheptanesulfonic acid	PFHpS	Perfluoroheptanesulfonic acid	PFHpS	
Perfluorooctanesulfonic acid	PFOS	Perfluorooctanesulfonic acid	PFOS	
Perfluorononanesulfonic acid	PFNS	Perfluorononanesulfonic acid	PFNS	
Perfluorodecanesulfonic acid	PFDS	Perfluorodecanesulfonic acid	PFDS	
Perfluorododecanesulfonic acid	PFDoS	Perfluorododecanesulfonic acid	PFDoDS	
1H ,1H ,2H ,2H -Perfluorohexane sulfonic acid	4:2FTS	4:2 Fluorotelomer sulfonate	4:2FTS	
1H ,1H ,2H ,2H -Perfluorooctane sulfonic acid	6:2FTS	6:2 Fluorotelomer sulfonate	6:2FTS	
1H ,1H ,2H ,2H -Perfluorodecane sulfonic acid	8:2FTS	8:2 Fluorotelomer sulfonate	8:2FTS	
Perfluorooctanesulfonamide	PFOSA	PFOSA	FOSA	
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	EtFOSAA	EtFOSAA	
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	MeFOSAA	MeFOSAA	
N-ethyl perfluorooctanesulfonamide	NEtFOSA	EtFOSA	EtFOSA	
N-methyl perfluorooctanesulfonamide	NMeFOSA	MeFOSA	MeFOSA	
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	EtFOSE	MeFOSE	
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	MeFOSE	EtFOSE	
Hexafluoropropylene oxide dimer acid	HFPO-DA	HFPO-DA (GenX)	HFPO-DA	
4,8-dioxa-3H-perfluorononanoic acid	ADONA	ADONA	ADONA	
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	9Cl-PF3ONS (F-53B Major)	9Cl-PF3ONS	
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	11Cl-PF3OUdS (F-53B Minor)	11Cl-PF3OUdS	
Perfluoro-3-methoxypropanoic acid	PFMPA	PFMPA	PFMPA	
Perfluoro-4-methoxybutanoic acid	PFMBA	PFMBA	PFMBA	
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	NFDHA	NFDHA	

Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	PFEESA	PFEESA	
3-Perfluoropropyl propanoic acid	3:3FTCA	3:3 Fluorotelomer carboxylate	3:3FTCA	
2H,2H,3H,3H-Perfluorooctanoic acid	5:3FTCA	5:3 Fluorotelomer carboxylate	5:3FTCA	
3-Perfluoroheptyl propanoic acid	7:3FTCA	7:3 Fluorotelomer carboxylate	7:3FTCA	
Perfluoro-n-[13C4]butanoic acid	13C4-PFBA	13C4-PFBA	13C4-PFBA	M4-PFBA
Perfluoro-n-[13C5]pentanoic acid	13C5-PFPeA	13C5-PFPeA	13C5-PFPeA	M5-PFPeA
Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid	13C5-PFHxA	13C5-PFHxA	13C5-PFHxA	M5-PFHxA
Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	13C4-PFHpA	13C4-PFHpA	13C4-PFHpA	M4-PFHpA
Perfluoro-n-[13C8]octanoic acid	13C8-PFOA	13C8-PFOA	13C8-PFOA	M8-PFOA
Perfluoro-n-[13C9]nonanoic acid	13C9-PFNA	13C9-PFNA	13C9-PFNA	M9-PFNA
Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid	13C6-PFDA	13C6-PFDA	13C6-PFDA	M6-PFDA
Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid	13C7-PFUnA	13C7-PFUnDA	13C7-PFUnDA	M7-PFUnDA
Perfluoro-n-[1,2-13C2]dodecanoic acid	13C2-PFDoA	13C2-PFDoDA	13C2-PFDoDA	M2-PFDoDA
Perfluoro-n-[1,2-13C2]tetradecanoic acid	13C2-PFTeDA	13C2-PFTeDA	13C2-PFTeDA	M2-PFTeDA
Perfluoro-1-[2,3,4-13C3]butanesulfonic acid	13C3-PFBS	13C3-PFBS	13C3-PFBS	M3-PFBS
Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid	13C3-PFHxS	13C3-PFHxS	13C3-PFHxS	M3-PFHxS
Perfluoro-1-[13C8]octanesulfonic acid	13C8-PFOS	13C8-PFOS	13C8-PFOS	M8-PFOS
1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexanesulfonic acid	13C2-4:2FTS	13C2-4:2FTS	13C2-4:2FTS	M2-4:2FTS
1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octanesulfonic acid	13C2-6:2FTS	13C2-6:2FTS	13C2-6:2FTS	M2-6:2FTS
1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decanesulfonic acid	13C2-8:2FTS	13C2-8:2FTS	13C2-8:2FTS	M2-8:2FTS
Perfluoro-1-[13C8]octanesulfonamide	13C8-PFOSA	13C8-FOSA	13C8-FOSA	M8-FOSA
N-ethyl-d5-perfluoro-1-octanesulfonamide	D5-NEtFOSA	d5-EtFOSA	d5-EtFOSA	M5-EtFOSA
N-methyl-d3-perfluoro-1-octanesulfonamide	D3-NMeFOSA	d3-MeFOSA	d3-MeFOSA	M3-MeFOSA
N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid	D5-NEtFOSAA	d5-EtFOSAA	d5-EtFOSAA	M5-EtFOSAA
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid	D3-NMeFOSAA	d3-MeFOSAA	d3-MeFOSAA	M3-MeFOSAA
N-methyl-d7-perfluorooctanesulfonamidoethanol	D7-NMeFOSE	d7-MeFOSE	d7-MeFOSE	M7-MeFOSE
N-ethyl-d9-perfluorooctanesulfonamidoethanol	D9-NEtFOSE	d9-EtFOSE	d9-EtFOSE	M9-EtFOSE
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	13C3-HFPO-DA	13C3-HFPO-DA	13C3-HFPO-DA	M3-HFPO-DA
Perfluoro-n-[2,3,4-13C3]butanoic acid	13C3-PFBA	13C3-PFBA	13C3-PFBA	
Perfluoro-n-[1,2,3,4-13C4]octanoic acid	13C4-PFOA	13C4-PFOA	13C4-PFOA	
Perfluoro-n-[1,2-13C2]decanoic acid	13C2-PFDA	13C2-PFDA	13C2-PFDA	
Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid	13C4-PFOS	13C4-PFOS	13C4-PFOS	
Perfluoro-1-hexane[18O2]sulfonic acid	18O2-PFHxS	18O2-PFHXS	18O2-PFHxS	
Perfluoro-n-[1,2-13C2]hexanoic acid	13C2-PFHxA	13C2-PFHXA	13C2-PFHxA	
Perfluoro-n-[1,2,3,4,5-13C5]nonanoic acid	13C5-PFNA	13C5-PFNA	13C5-PFNA	

Table 5. Acceptance QC limits for wastewater and soil samples

Compounds	iDOC		BS/BS-LL Recovery (%)
	Recovery (%)	RSD (%)	
PFBA	70-135	21	70-140
PFPeA	70-135	23	65-135
PFHxA	70-135	24	70-145
PFHpA	70-135	28	70-150
PFOA	65-155	27	70-150
PFNA	70-140	28	70-150
PFDA	65-140	26	70-140
PFUnA	70-135	29	70-145
PFDoA	70-130	21	70-140
PFTrDA	60- 145	29	65-140
PFTeDA	70-145	27	60-140
PFBS	70-140	23	60-145
PFPeS	70-135	25	65-140
PFHxS	70-135	27	65-145
PFHpS	70-140	30	70-150
PFOS	70-140	29	55-150
PFNS	70-135	29	65-145
PFDS	70-135	30	60-145
PFDoS	45-135	35	50 – 145
4:2FTS	70 – 135	27	70-145
6:2FTS	70-135	32	65-155
8:2FTS	70-140	33	60-150
PFOSA	70-135	22	70-145
NMeFOSA	70-135	30	60-150
NEtFOSA	70-130	26	65 – 145
NMeFOSAA	65-140	32	50-140
NEtFOSAA	70-135	28	70-145
NMeFOSE	70-135	29	70-145
NEtFOSE	70-130	21	70-135
HFPO-DA	70 – 135	23	70-140
ADONA	70-135	23	65-145
PFMPA	60-140	23	55-140
PFMBA	65 – 145	27	60-150
NFDHA	65-140	37	50-150
9Cl-PF3ONS	70-145	30	70-155

	iDOC		
Compounds	**Recovery (%)**	**RSD (%)**	**BS/BS-LL Recovery (%)**
11Cl-PF3OUdS	50-150	35	55-160
PFEESA	70-135	25	70-140
3:3FTCA	70-130	23	65-130
5:3FTCA	70 – 130	24	70-135
7:3FTCA	55 – 130	34	50 – 145

Table 6. QC Acceptance Limits for EIS and NIS Recoveries in wastewater and soil samples

EIS Compound	Recovery Range (%)
$^{13}C_4$-PFBA	5 – 130 *
$^{13}C_5$-PFPeA	40-130
13C5-PFHxA	40-130
$^{13}C_4$-PFHpA	40-130
$^{13}C_8$-PFOA	40-130
$^{13}C_9$-PFNA	40-130
$^{13}C_6$-PFDA	40-130
$^{13}C_7$-PFUnA	30 – 130
$^{13}C_2$-PFDoA	10 – 130
$^{13}C_2$-PFTeDA	10 – 130
$^{13}C_3$-PFBS	40-135
$^{13}C_3$-PFHxS	40-130
$^{13}C_8$-PFOS	40-130
$^{13}C_2$-4:2FTS	40 – 220
$^{13}C_2$-6:2FTS	40 – 200
$^{13}C_2$-8:2FTS	40-300
$^{13}C_8$-PFOSA	40 – 130
D_3-NMeFOSA	10 – 130
D_5-NEtFOSA	10 – 130
D_3-NMeFOSAA	35-170
D_5-NEtFOSAA	25-135
D_7-NMeFOSE	10 – 130
D_9-NEtFOSE	10 – 130
$^{13}C_3$-HFPO-DA	40-130
13C3-PFBA	50-200
13C2-PFHxA	
13C4-PFOA	
13C5-PFNA	
13C2-PFDA	
18O2-PFHxS	
13C4-PFOS	

* Recovery of 13C4-PFBA can be problematic in some field samples. Although the lower limit for recovery for this EIS is set below 10%, laboratories should routinely track recovery of this EIS and take reasonable steps to ensure that recovery is at least 10% in the majority of samples.

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
Reference,15.1	Reference updated to the most current method reference.
2.1	Updated the method reference.
Table 6	Recovery limits for $^{13}C_2$-4:2FTS and D_3-NMeFOSAA is revised according to the EPA method 1633

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
00	01/05/2023	Anita Jagtap
01	05/22/2023	Olga Azarian
02	06/20/2023	Olga Azarian
03	07/21/2023	Olga Azarian
04	8/8/2023	Olga Azarian
05	01/04/2023	Olga Azarian
06	2/05/2024	Olga Azarian
07	03/21/2024	Raj Singh

END OF DOCUMENT

LAB SUPERVISOR: Bridget Kelley

QA OFFICER: Olga Y. Agoian

EFFECTIVE DATE: 4-25-23

TITLE: METHOD 8260D, VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (GC/MS)
REFERENCES: SW846 8260D (Revision 4, June 2018), SW846 8000D (Revision 5, March 2018)
REVISED SECTIONS: 10.4.1, 11.3.2, 12.1
DELETED SECTIONS: 11.3.3, 11.3.4

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the analytical procedures, which are utilized by SGS to acquire samples for analysis of volatile organic compounds by gas chromatographic/mass spectrometric (GC/MS) following purge and trap utilizing the internal standard technique. The compounds in Table 1 may be determined by this method. An option has been included for the analysis of 1,4-Dioxane by selected ion monitoring GC/MS (GC/MS-SIM).

1.2 This analytical method is designed for nearly all types of samples, regardless of water content, including ground water, aqueous sludges, liquors, waste solvents, oily wastes, tars, filter cakes, sediments and soils.

1.3 The applicable concentration range of this method is compound, matrix, and instrument dependent. Volatile water-soluble compounds can be included in this analytical technique. However, for some low-molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides, quantitation limits are approximately ten times higher because of poor purging efficiency. Determination of some structural isomers (i.e. xylenes) may also be hampered by coelution.

2.0 SUMMARY OF METHOD

2.1 Volatile compounds are introduced into the gas chromatograph by purge-and-trap (Method 5030/5035). Method 5030 may be used directly on ground water samples. Method 5035 is used for low-concentration and medium-concentration soils, sediments, and wastes. Medium concentration samples are preserved and stored in methanol prior to purge-and-trap analysis.

2.2 An inert gas is bubbled through a 5 ml sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic (GC) column.

2.3 The volatile compounds are separated by the temperature programmed GC column and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.4 The peaks detected are qualitated by comparison to characteristic ions and retention times specific to the known target list of compounds.

2.5 Once identified the compound is quantitated by comparing the response of major (quantitation) ion relative to an internal standard technique with an average response factor generated from a calibration curve.

2.6 Additional unknown peaks with a response > 10 % of the closest internal standard may be processed through a library search with comparison to a database of approximately 75,000 spectra. An estimated concentration is quantitated by assuming a response factor of 1.

2.7 Water soluble volatile organic and other poor purging compounds maybe analyzed using this methodology, however this method is not the method of choice for these compounds and the laboratory's ability to achieve all calibration and quality control criteria for this method cannot be guaranteed. These compounds are noted as (pp) in Table 7.

2.8 The method includes an analytical option for the analysis of 1,4-Dioxane by GC/MS-SIM. The selected ions that are characteristic of the analytes of interest are analyzed using lower concentrations of calibration standards under the same MS conditions. SIM analysis is performed upon client request and is documented in the report.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve and may vary depending on matrix interferences, sample volume or weight and percent moisture. Detected concentrations below this concentration cannot be reported without qualification. See Table 10.

3.1.1 Compounds detected at concentrations between the reporting limit and MDL are quantitated and qualified as "J", estimated value. Program or project specifications may dictate that "J" qualified compounds are not to be reported.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B, revision 2. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method.

3.2.2 Process all raw data for the replicate analysis in each MDL study. Forward the processed data to the QA group for archiving.

3.2.3 Calculated MDLs may not be feasible in the analysis of samples, particularly in regard to compounds in table 11 and common laboratory solvents (methylene chloride and acetone). In these cases, the MDLs may be raised from the calculated value to a maximum of half the LOQ to avoid false positives being reported.

4.0 DEFINITIONS

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank, Instrument Blank, Storage Blank, Cleanup Blank and Sulfur Blank.

4-BROMOFLUOROBENZENE (BFB) - the compound chosen to establish mass spectral instrument performance for volatile (VOA) analyses.

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CONTINUING CALIBRATION - analytical standard run every 12 hours to verify the initial calibration of the system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - used herein synonymously with the terms continuous extraction, continuous liquid extraction, and liquid extraction. This extraction technique involves boiling the extraction solvent in a flask and condensing the solvent above the aqueous sample. The condensed solvent drips through the sample, extracting the compounds of interest from the aqueous phase.

EXTRACTED ION CURRENT PROFILE (EICP) - a plot of ion abundance versus time (or scan number) for ion(s) of specified mass (Es).

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer to the target compounds.

INTERNAL STANDARDS - compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatiles), and sample extract (for semivolatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is <u>not</u> synonymous with phase (liquid or solid).

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD DETECTION LIMITS (MDLs) - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is distinguishable from method blank results.

PERCENT DIFFERENCE (%D) - As used in this SOP and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

PRIMARY QUANTITATION ION - a contract specified ion used to quantitate a target analyte.

REAGENT WATER - water in which an interferant is not observed at or above the minimum detection limit of the parameters of interest.

RECONSTRUCTED ION CHROMATOGRAM (RIC) - a mass spectral graphical representation of the separation achieved by a gas chromatograph: a plot of total ion current versus retention time.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

INSTRUMENT BLANK – a system evaluation sample containing lab reagent grade water with internal standards and surrogate standards added. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Plan and Personal Protection Policy, which include the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the

chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 The following analytes covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichlorethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Primary standards of these toxic compounds must be prepared in a hood. A NIOSH/Mass approved toxic gas respirator must be worn when the analyst handles high concentrations of these toxic compounds.

6.0 INTERFERENCES

6.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

6.2 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device must be avoided.

6.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

6.4 Contamination by carry-over can occur whenever high level and low-level samples are sequentially analyzed.

6.4.1 Whenever an unusually concentrated sample is encountered, it must be followed by an analysis of an instrument blank to check for cross contamination. Refer to Table 11 for compounds that may cause carryover for this method.

6.4.2 It may be necessary to wash the purging device with methanol, rinse it with organic-free water, and then dry the purging device in an oven at 105^0 C. Follow the instrument manual for instructions on cleaning. Document the occurrence in the maintenance log and notify the manager/supervisor.

6.4.2.1 Clean and bake purging tube.

6.4.2.2 Clean or replace purge needle.

6.4.2.3 Clean and bake sample filter or sparge filter.

6.4.2.4 Clean and bake sample loop.

6.4.2.5 Replace trap if necessary.

6.4.2.6 Replace water management module if necessary.

6.4.2.7 Rinse transfer line with methanol. Caution: disconnect the trap before rinsing.

6.4.3 In extreme situations, the entire purge-and trap device may require dismantling and cleaning. Follow the instrument's manual for instructions on disassembly. Document the occurrence in the maintenance log and notify the manager/supervisor. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples.

6.4.4 If the contamination has been transferred to gas chromatograph, any of the following approaches may be used to cleanup the instrument.

6.4.4.1 Baking out the column between analyses.

6.4.4.2 Change the injector liner to reduce the potential for cross-contamination.

6.4.4.3 Remove a portion of the analytical column in the case of extreme contamination.

6.4.5 The oven temperature program must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are stripped from the chromatographic column.

6.5 Special precautions must be taken during the analysis to avoid contamination from methylene chloride and other common laboratory solvents.

6.5.1 The sample storage and analytical area must be isolated from all atmospheric sources of methylene chloride or other common solvents.

6.5.2 Laboratory clothing worn by the analyst must be clean and used in designated areas only. Clothing previously exposed to solvent vapors in the organics sample preparation laboratory can contribute to sample contamination.

6.6 Samples with suspected or known permanganate levels must be preserved with ascorbic acid at collection. The purpose of the ascorbic acid is to remove the permanganate which is an oxidizer. There is potential that the analytes of concern will undergo an oxidative transformation which would no longer be representative of the concentrations as the site.

7.0 SAMPLE HANDLING AND PRESERVATION AND HOLDING TIME

7.1 HANDLING and PRESERVATION

7.1.1 Water samples

7.1.1.1 Container - 40 ml glass screw-cap VOA vial with Teflon-faced silicone septum. The 40-ml glass VOA vials are pre-cleaned and certified.

7.1.1.2 Acrolein & Acrylonitrile

7.1.1.2.1 If acrolein and acrylonitrile are to be analyzed, collect 3, 40 mL VO vials of sample unpreserved. Samples for acrolein and acrylonitrile analysis receiving no pH adjustment must be analyzed within 7 days of

sampling. All samples must be footnoted stating samples were unpreserved and analyzed within 7 days.

7.1.1.3 Collect all samples in triplicate. Test all samples for residual chlorine using test paper for free and total chlorine. If samples contain residual chlorine, three milligrams of sodium thiosulfate must be added for each 40 ml of water sample.

7.1.1.4 Fill sample bottles to overflowing, but do not flush out the dechlorinating agent. Sample must be taken with care so as to prevent any air or bubbles entering vials creating headspace.

7.1.1.5 Adjust the pH of all samples to ≤ 2 at the time of collection, but after dechlorination, by carefully adding two drops of 1:1 HCl for each 40 ml of sample. Seal the sample bottles, Teflon face down, and mix for one minute. Or VOA vials containing the preservative (HCL) may be used.

Note: Do not mix the sodium thiosulfate with the HCl in the sample bottle prior to sampling.

7.1.1.6 The samples must be protected from light and refrigerated at 0 - ≤ 6 °C from the time of receipt until analysis.

7.1.1.7 An alternate preservative that may be used when suspected or known levels of permanganate exist in a sample is 25 mg of ascorbic acid per 40 ml vial.

7.1.1.7.1 Ascorbic acid is added to remove the permanganate which is an oxidizer.

7.1.1.7.2 Fill the sample bottles to overflowing, but do not flush out the ascorbic acid.

7.1.1.7.3 The samples must be protected from light and refrigerated at 0 - ≤ 6 °C from the time of receipt until analysis.

7.1.2 Soil Samples

7.1.2.1 Refer to the SOP for SW846 Method 5035 for preservation requirement of non-aqueous solids.

7.2 HOLDING TIME

7.2.1 Water Samples.

7.2.1.1 All samples are to be analyzed within 14 days of sampling (HCl preserved for aqueous sample) unless otherwise specified by the contract. The sample preservation deficiency is noted in the analytical run logbook when the analyst checks the pH at the bench. If the pH is not <2, the analyst notifies the supervisor, who then notifies Client Service Dept. A comment is added to the result page and Non-Conformance Summary.

7.2.1.2 Acrolein & Acrylonitrile

7.2.1.2.1 Samples for acrolein and acrylonitrile analysis receiving no pH adjustment must be analyzed within 7 days of sampling.

7.2.2 Soil Samples

7.2.2.1 Refer to the SOP for SW846 Method 5035 for holding time requirement of non-aqueous solids.

7.2.2.2 All samples are analyzed within 14 days of sampling unless otherwise specified.

8.0 APPARATUS AND MATERIALS

8.1 SYRINGE

8.1.1 10, 25, 50, 100, 250, 500, 1000 and 5000 µl graduated syringes, held manually (Hamilton/equiv.).

8.1.2 5 ml and 50 ml glass gas tight syringes with Luerlok end, if appropriate for the purging device.

8.2 BALANCE

8.2.1 Analytical balance capable of weighing 0.0001 gram.

8.2.2 Top loading balance capable of weighing 0.1 gram.

8.3 PURGE AND TRAP DEVICES

8.3.1 The autosampler models are used for purging, trapping and desorbing the sample into GC column.

- O.I. Model 4560 sample concentrator with 4551 vial multi-sampler
- O.I. Model 4560 sample concentrator with 4552 Water/Soil multi-sampler
- EST Analytical Encon Evolution concentrator with EST Centurion multi-sampler

8.3.2 The sample purge vial must be designed to accept 5 ml of sample with a water column at least 3 cm deep.

8.3.3 The auto-sampler is equipped with a heater capable of maintaining the purge chamber at 40 °C to improve purging efficiency. The heater is to be used for low level soil/sediment analysis, but not for water or medium level soil/sediment analysis.

8.3.4 The OI #10 trap is 42 cm with an inside diameter of 0.105 inches. The trap must be packed to contain the following absorbents (3-ring) and must be conditioned at 210 °C for 30 minutes by backflushing with a Helium or Nitrogen gas flow at least 20 ml/min before initial use.

- Tenax (2,6-Diphenylene oxide polymer).

- Silica gel.
- Carbon Molecule Sieve (CMS).

8.3.5 The EST K trap for EST instruments: Carboack B/Carboxen 1000 &1001 with recommended conditions as follows:

Temperature (°C)/Time (minutes)

Desorb	Bake	Precondition
250/0.5-6	260/4	270/60

8.3.6 The desorber must be capable of rapidly heating the trap to 190^0 C for desorption. Do not exceed 210^0 C during bake-out mode. Alternatively, follow manufacturer's instructions.

8.4 GAS CHROMATOGRAPH/MASS SPECTROMETER SYSTEM

8.4.1 Gas Chromatograph.

8.4.1.1 An analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases.

8.4.1.2 The injection port must be suitable for split or splitless with appropriate interface.

8.4.1.3 The narrow bore capillary column is directly coupled to the source for HP-6890/7890 or Agilent 6890/7890 model.

8.4.1.4 The wide bore capillary column is interfaced through a jet separator to the source for HP-5890 model.

8.4.2 Column.

- 75 m x 0.53mm ID x 3 μm film thickness capillary column coated with DB-624 (J&W Scientific), or equivalent. Condition as per manufactures directions.
- 105 m x 0.53mm ID x 3 μm film thickness capillary column coated with HP-VOA, or equivalent. Condition as per manufactures directions.
- 60 m x 0.25mm ID x 1.4 μm film thickness capillary column coated with DB-624 (J&W Scientific), or equivalent. Condition as per manufactures directions.
- 60 m x 0.45mm ID x 1.7 μm film thickness capillary column coated with DB-VRX (J&W Scientific), or equivalent. Condition as per manufactures directions.
- 30 m x 0.25mm ID x 1.4 μm film thickness capillary column coated with RTX-624, or equivalent. Condition as per manufactures directions.
- 20 m x 0.25mm ID x 1.4 μm film thickness capillary column coated with RTX-624, or equivalent. Condition as per manufactures directions.

8.4.3 Mass Spectrometer.

8.4.3.1 HP5973, HP5970 Agilent 5973, or Agilent 5975 is capable of scanning from 35 to 270 amu every 2 seconds or less, utilizing 70-volt (nominal) electron energy in the electron impact ionization mode.

8.4.3.2 The mass spectrometer must be capable of producing a mass spectrum which meets all the criteria in Table 3 when injecting or purging 50 ng of the GC/MS tuning standard - Bromofluorobenzene (BFB).

8.4.3.3 SIM Mode – Capable of selective ion grouping at specified retention times for applications requiring quantitation limits below the normal range of electron impact mass spectrometry or for increased compound sensitivity (Table 2a).

8.5 DATA SYSTEM

8.5.1 Data Acquisition and Instrument Control (HP Chemstation) **-** A computer system is interfaced to the mass spectrometer, which allows the continuous acquisition and storage on a machine-readable media (disc) of all mass spectra obtained throughout the duration of the chromatographic program.

8.5.2 Data Processing (HP Enviroquant) - The software accommodates searching of GC/MS data file for target analytes which display specific fragmentation patterns. The software also allows integrating the abundance of an EICP between specified time or scan number limits. The data system includes the recent version of the EPA/NBS or NIST98 mass spectral library for qualitative searches of non-target compounds present in the chromatogram. The data system flags all data files that have been edited manually by laboratory personnel.

8.5.3 Off-line Magnetic Tape Storage Device (Lagato Networker) - The magnetic tape storage device copies data for long-term, off-line storage.

9.0 REAGENTS AND STANDARDS

9.1 Solvent

9.1.1 Methanol: purge-and-trap grade quality or equivalent. Store separately, away from the other solvents.

9.2 Reagent Water

9.2.1 Reagent water is defined as water in which an interferant is not observed at the method detection limit of the parameters of interest.

9.2.2 Reagent water is generated by either passing tap water through a bed of approximately one pound of activated carbon or by using the water purification system at SGS that is a series of deionizers and carbon cartridges.

9.3 Stock Standard Solutions

9.3.1 Commercially prepared standards used.

9.3.1.1 EPA Method 524.2 Volatiles (78 components): Absolute (or equivalent) at 200 µg/ml or 2,000 µg/ml concentration.

9.3.1.2 Custom Volatiles Mix A: Restek (or equivalent) at 2,000 µg/ml concentration.

9.3.1.3 Custom Volatiles Mix B: Restek (or equivalent) at 2,000 - 100,000 µg/ml concentration.

9.3.1.4 VOC Gas Mixture: Restek (or equivalent) contains 200 µg/ml or 2,000 µg/ml of the following compound**s** in methanol.

- Bromomethane
- Chloroethane
- Chloromethane
- Dichlorodifluoromethane
- Trichlorofluoromethane
- Vinyl Chloride

9.3.1.5 Multiple neat compounds and individual compounds at varying concentrations in methanol.

9.3.1.6 Surrogate standard mixture: Absolute (or equivalent) at a concentration of 10,000 µg/ml each surrogate compound.

- 1,2-Dichloroethane-d_4
- Dibromofluoromethane
- Toluene-d_8
- 4-Bromofluorobenzene

9.3.1.7 Internal standard mixture: Sigma-Aldrich (or equivalent) neat compounds diluted to 20,000 µg/ml except Tert Butyl Alcohol-d_9, which is diluted to a concentration of 50,000 µg/ml. The following five internal standards are used that exhibit similar analytical behavior to the compounds of interest.

- 1,4-Dichlorobenzene-d_4
- 1,4-Difluorobenzene
- Chlorobenzene-d_5
- Pentafluorobenzene
- Tert Butyl Alcohol-d_9

9.3.1.8 1,4-Dioxane Solution for SIM: Absolute (or equivalent) at 100 µg/ml in methanol.

9.3.1.9 Ketones mixture: Absolute (or equivalent) at 20,000 µg/ml for Acetone, 2-Butanone, 4-methyl-2-pentanone (MIBK), and 2-hexanone prepared at concentrations 300 ug/ml for soil matrix and 300 ug/ml for aqueous matrix.

9.3.2 Unopened stock standard (ampoules) must be stored according to manufacturer's documented holding time and storage temperature recommendations (usually placed on the ampoule).

9.3.3 After opened, stock standards, internal standards, and surrogate solutions must be replaced after 6 months (one month for purgeable gases standard) or sooner if manufacture expiration date come first or comparison with quality control check samples indicates degradation.

9.3.4 Store all stock standards in vials with minimal headspace and Teflon lid liners after open, protect from light, and refrigerate to –10°C or colder or as recommended by the standard manufacturer.

9.3.5 Return the standards to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

9.4 Internal Standard and Surrogate Solution

9.4.1 Five internal standard and surrogate spiking solutions are prepared in methanol per Table 8.A.

9.4.1.1 25 μg /ml internal standard and surrogate mixture.

9.4.1.2 250 μg /ml internal standard and surrogate mixture.

9.4.1.3 100 μg/ml surrogate mixture.

9.4.1.4 25 μg /ml internal standard mixture.

9.4.1.5 250 μg /ml internal standard mixture.

9.4.2 A calibration range must be constructed for the surrogate compounds. Accordingly, appropriate amounts of surrogates are mixed with each calibration solution to define a range similar to the target compounds.

9.4.3 Each 5 ml sample, QC sample, and blank undergoing analysis must be spiked with any one of the above spiking solutions (depending upon the type of standards addition modules used), resulting in a concentration of 50 μg/l of each compound.

9.4.4 Prepare fresh internal standard and surrogate spiking solutions every six months, or sooner, if manufacturer's expiration dates come first or if the solution has degraded or evaporated.

9.5 Secondary Dilution Standards

9.5.1 Using stock standard solutions prepare secondary dilution standards in methanol containing the compounds of interest, either singly or mixed together.

9.5.1.1 100 μg/ml V8260 (ABK) mixture: prepared from 2,000 μg/ml stock solution and mixtures from neat compounds prepared in-house. (see Table 8-C)

9.5.1.2 100 μg/ml V8260 Acrolein: prepared from 90-95% neat compound. (see Table 8-C)

9.5.1.3 100 μg/ml Gas mixture: prepared from 2,000 μg/ml stock solution. (see Table 8-C)

9.5.2 Replace after one month for non-gas mixtures (one week for gas mixtures) or sooner if manufacture expiration date come first or comparison with quality control check samples indicates degradation.

9.5.3 Store all secondary dilution standards in vials with no headspace and Teflon lid liners, protect from light, and refrigerate to – 10°C or colder or according to manufacturer's storage temperature recommendations.

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9.5.4 Return the standards to the freezer as soon as preparation is finished to prevent the evaporation of volatile compounds.

9.6 Aqueous Calibration Standard Solutions

9.6.1 Initial Calibration Standards

9.6.1.1 Prepare a minimum of five aqueous calibration standard solutions containing the surrogate compounds as Table 8-D.1 or 8-D.2.

9.6.1.2 To prepare a calibration standard, add a measured volume of secondary dilution standard solutions and the surrogate spiking solution to an aliquot of reagent water in the flask. Use a micro-syringe and rapidly inject the methanol standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Bring to volume. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask.

9.6.1.2.1 1,4-Dioxane for SIM analysis is prepared from primary stock standard (100ppm).

9.6.2 Continuing Calibration Standard

9.6.2.1 A continuing calibration standard at a concentration of 50 μg/l is prepared as the scheme outlined in Table 8-E.

9.6.3 Aqueous standards are not stable and may be stored up to 24 hours if held in Teflon sealed screw-cap vials with zero headspace at 4°C (± 2°C). Protect the standards from light. If not so stored, they must be discarded after use, unless they are set up to be purged by an autosampler.

9.6.4 When using an autosampler, standards may be retained up to 12 hours if they are in purge tubes connected via the autosampler to the purge and trap device.

9.7 Second Source Calibration Check Standard (ICV)

9.7.1 Prepare the second source calibration check standards from separate manufacturer source or from a manufacturer's batch prepared independently from the batch used for calibration curve following the procedures in Section 9.6. At a minimum, an ICV must be analyzed with every initial calibration.

9.7.2 For 1,4-Dioxane via SIM: Prepare the second source calibration check standard using 20 µl of a 100ppm (Absolute or equivalent) to 100 mL of reagent water which yields a 20 ppb standard.

9.8 4-Bromofluorobenzene (BFB) Standard

9.8.1 Two BFB solutions are prepared in methanol per Table 8-B.

9.8.1.1 25 µg /ml solution for direct injection.

9.8.1.2 250 µg /ml solution for purging.

9.8.2 The solution must be replaced after 6 months or sooner if mass spectrum indicates degradation or if manufacture expiration date comes first.

9.9 Ascorbic Acid

10.0 CALIBRATION

10.1 Daily Maintenance. Routine Daily maintenance must be performed before any tuning, calibration or sample analysis activities are initiated. These include checks of the following items:

Purge and Trap Device:

Clean & bake purge tube
Bake trap and transfer lines
Check or refill internal/surrogate spike solution on SIM/SAM vials
Clean/replace syringe (if necessary)
Change and refill rinse bottle
Empty and rinse waste bottle

GC Oven: (if necessary)

Change septum
Change liner
Clip column, indicated by carbon build-up

10.2 Initial Calibration

10.2.1 The calibration range covered for routine analysis under RCRA, and SIM, employs standards of 0.2, 0.5, 1(specified compounds only), (2)*, 5, 10, 20, 50, 100, 200,(300 or 400)* µg/l. (*instrument dependent). Optionally 4 and 8 ug/l standards may replace the 5 and 10 ug/l standards. A minimum of five standards must be run sequentially. The low calibration standard defines the reporting limit. Lower concentration standards (0.2, 0.5, 1.0 or 2.0 µg/l) may be needed to meet the reporting limit requirements of state specific regulatory programs. Refer to Table 8-D-1 and 8-D-2 for calibration standard preparation.

10.2.2 The surrogates are introduced to the calibration standards automatically by the autosampler. For this calibration option the surrogate linear response is less important, since multiple concentrations of surrogates are not being measured. Instead, the surrogate concentration remains constant throughout and the recovery of this known concentration can easily be attained without demonstrating if the response is linear.

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10.2.2.1 Optional: The surrogates can be added manually. In order to compensate for the difference between the automatic and manual surrogate additions a correction factor must be applied to the amount of surrogate added in Table 8-D. To determine the correction factor divide the surrogate concentration from an automatic injection by the surrogate concentration from a manual injection for each of the surrogates. Average the result for each of the surrogates to determine the correction factor. Finally multiply the correction factor by the appropriate amount of surrogate from Table 8-D and add this amount to the standard.

10.2.3 For water and medium-level soil calibration: Transfer and fill up (no air space) each standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray.

10.2.4 For low-level soil calibration: Transfer 5 ml of each standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray.

10.2.4.1 When calibrating for Method 5035 low-level samples, if the sodium bisulfate option was used, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of each standard into vial otherwise do not add sodium bisulfate. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds. Cap the vial with Teflon septum and place it into O.I sample tray.

10.2.5 The linear range covered by this calibration is the highest concentration standard.

10.2.6 Program the autosampler to add internal standard mixture (and optionally surrogate) to each standard. This results in a concentration of 50 μg/l for each internal standard (and surrogate).

10.2.6.1 For O.I. SIM spiker: Automatically adds 5 μl of 50 μg/ml internal standard solution **(**Section 9.4.1.4) or Internal Standard/Surrogate solution (Section 9.4.1.1) to each standard.

10.2.6.2 For O.I. SAM spiker: Automatically adds 1 μl of 250 μg/ml internal standard solution (Section 9.4.1.5) or Internal Standard/Surrogate solution Section 9.4.1.2) to each standard.

10.2.7 Analyze the standard solutions using the conditions established in Section 11.0. Whenever the highest concentration standard is analyzed, it is usually followed by the analyses of two reagent water blanks. Further analysis may not proceed until the blank analysis is demonstrated to be free of interferences.

10.2.8 Each analyte is quantitatively determined by internal standard technique using the closest eluting internal standard and the corresponding area of the major ion. See Table 7.

10.2.9 The Response Factor (RF) is defined in Section 13.1. Calculate the mean RF for each target analyte using minimum of five RF values calculated from the initial calibration curve.

10.2.10 For the initial calibration to be valid, the following criteria must be met.

10.2.10.1 The percent relative standard deviation (% RSD) (see Section 13.2) of all target analytes must be less than or equal to 20%.

10.2.10.2 If the average response factor criteria cannot be achieved, and if the problem is associated with one or more of the standards, reanalyze the standards and recalculate the RSD. The instrument logbook must have clear documentation as to what the suspected problem was.

10.2.10.3 Alternately, if the average response factor criteria cannot be achieved, the calibration range can be narrowed by dropping the low or high point of the curve. Multiple levels may be removed, but removal of interior levels is not permitted

10.2.10.3.1 The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end.

10.2.10.3.2 The laboratory may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. If a calibration standard was removed from the interior of the calibration, this particular standard calibration level must be removed for all analytes. Removal of calibration points from the interior of the curve is not to be used to compensate for lack of maintenance or repair to the instrument.

10.2.10.3.2.1 The laboratory must adjust the LOQ/reporting limit and quantitation range of the calibration based on the concentration of the remaining high and low calibration standards.

10.2.10.3.2.2 The laboratory must ensure that the remaining initial calibration standards are sufficient to meet the minimum requirements for number of initial calibration points as mandated by the method, or regulatory requirements.

10.2.10.3.2.3 The laboratory may replace a calibration standard provided that:

a) the laboratory analyzes the replacement standard within twenty-four (24) hours of the original calibration standard analysis for that particular calibration level;

b) the laboratory replaces all analytes of the replacement calibration standard if a standard within the interior of the calibration is replaced;
c) the laboratory limits the replacement of calibration standards to one calibration standard concentration.

10.2.10.3.2.4 The laboratory must document a technically valid reason for either removal or replacement of any interior calibration point.

10.2.10.4 If the average response factor criteria still cannot be achieved, employ an alternative calibration linearity model. Specifically, linear regression using a least squares approach may be employed.

10.2.10.4.1 If linear regression is employed select the linear regression calibration option of the mass spectrometer data system. Forcing the calibration model through the origin (for analytes that are consistently detected in the blanks) allows for a better estimate of the background level of blank contaminants. An accurate estimate of background contamination is necessary to set method reporting limits for analytes when blank levels are problematic.

10.2.10.4.2 The correlation coefficient (r value) must be ≥0.99 for each compound to be acceptable. The calculation of relative error (%RE) must be performed for two calibration levels (near the mid-point of the initial calibration and the standard at the lowest level):

$$\%\ Relative\ Error = \frac{x'_i - x_i}{x_i} \times 100$$

x_i = True value for the calibration standard
x'_i = Measured concentration of the calibration standard

10.2.10.4.2.1 When calculating the calibration curves using the linear regression model, two quantitation checks must be performed by re-fitting the response from the standard at or near the mid-point of the initial calibration and the standard at the lowest level back into the curve.

10.2.10.4.2.2 The recalculated concentration of these two quantitation checks must be within ± 30% of the standard's true concentration for mid-point and ±50% for lowest level.

10.2.10.5 The initial calibration criteria for this method apply to all additional compounds of concern specified by the client.

10.2.10.6 If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient for the linear calibration option, then the chromatographic system is considered too reactive for the analysis to begin. Perform corrective action and recalibrate if the calibration criteria cannot be achieved.

10.2.10.7 A quadratic calibration model is allowed if the linear regression fails.

10.2.10.7.1 This may only be used for historically poor performing compounds (e.g. ketones).

10.2.10.7.2 A minimum of six calibration points is required. Do not employ as a calibration point. 0,0

10.2.10.7.3 Quadratic calibration models cannot be used to extend the calibration range.

10.2.10.7.4 The calculation of relative error (%RE) must be performed for two calibration levels- the standard at or near the mid-point of the initial calibration and the standard at the lowest level. The relative error at the lowest level must be within ± 50% of the standard's true concentration, and at the mid-point it must be ±30% of the true concentration.

10.2.10.8 It is recommended that the minimum response factor for the most common target analytes in table 12 must be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Poor purging compounds such as ketones may not meet the recommendations.

10.3 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

10.3.1 The calibration is verified with a calibration check standard at 50 µg/l from an external source (Section 9.7). It must be analyzed immediately following the initial calibration.

10.3.2 The percent difference (% D) (Section 13.3) for this standard must meet the criteria of 30% for all the target compounds.

10.3.2.1 If % D is greater than 30%, reanalyze the second source check. If the criteria cannot be met upon re-injection, re-prepare the second source solution using a fresh ampoule and repeat the process.

10.3.2.2 If the %D criteria cannot be achieved after re-preparation of the second source, prepare a third source and repeat the process. Make fresh calibration standards using one of the two standard sources that match each other and repeat the initial calibration.

10.4 Continuing Calibration Verification Standard (CCV)

10.4.1 A continuing calibration verification standard at a concentration near mid-level of the initial calibration range (50 µg/l) must be acquired every 12 hrs or at the beginning of each analytical batch. If samples are analyzed within twelve hours of initial calibration, the CCV may be omitted, and injection of the last ICal standard may be used as the starting time reference for evaluation.
NOTE: The 12-hour clock starts at the time of injection of daily CCV.

10.4.1.1 For water and medium level soil analysis: Transfer and fill up (no air space) the calibration verification standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray. Analyze as per Section 11.7.

10.4.1.1.1 Vary the concentration of the continuing calibration verification standard on alternate verifications (i.e. every other calibration verification) using an alternative concentration standard. The standard selected must be lower than the midpoint calibration standard.

10.4.1.2 For low-level soil analysis: Transfer 5 ml of the calibration verification standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray. Analyze as per Section 11.7.

10.4.1.2.1 When calibrating for Method 5035 low-level samples, if the sodium bisulfate option was used add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of the calibration verification standard into vial, otherwise do not use sodium bisulfate. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds. Analyze as per Section 11.7.

10.4.1.3 A continuing calibration standard is analyzed whenever the analyst suspects that the analytical system is out of calibration. If the calibration cannot be verified, corrective action is performed to bring the system into control. Analysis may not continue until the system is under control.

10.4.2 For the continuing calibration to be valid, all of the following specified criteria must be met.

10.4.2.1 Each of the most common target analytes in the calibration verification standard must meet the minimum response factors as noted in Table 12.
This criterion is particularly important when the common target analytes are also critical project-required compounds. This is the same check that is applied during the initial calibration.

10.4.2.2 All target compounds of interest must be evaluated using a 20%variability criterion. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid.

10.4.2.3 Due to the large numbers of compounds that may be analyzed by this method, some compounds will fail to meet the criteria. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples.

10.4.2.4 In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values.

10.4.2.4.1 Compounds with response factors that exceed the 20% D in the CCV compared to the initial calibration with high bias may only be reported as an estimated value.

10.4.2.4.2 Compounds that do not meet the 20% D in the CCV compared to the initial calibration due to low response factors can only be reported if the low sensitivity of the instrument is still achieved. This sensitivity must be verified by running a low-level standard check at the RL. If a positive result for the compound is found, then adequate sensitivity has been demonstrated and the run can proceed. Non-detect results for samples may be reported, positive results, if reported, must be done as an estimated value.

10.4.3 If the first continuing calibration verification (CCV) does not meet criteria, a second standard can be analyzed immediately or after the corrective action was performed. If the second CCV fails to meet criteria, then corrective actions must be performed. Such as: auto-tuning, routine system cleaning and routine system maintenance. Notify the team leader/manager.

10.4.3.1 If the second CCV trial fails, the lab must demonstrate acceptable performance after corrective action with two consecutive passing calibration verifications (CCVs) OR a new initial calibration. The Instrument Logbook and Maintenance Logbook must have clearly documented notations as to what the problem was and what corrective action was implemented.

10.4.3.1.1 If the lab has not verified calibration, samples cannot be analyzed.

10.4.3.1.2 However, in the case where samples are analyzed on the system where the CCV does not meet the criteria the data must be flagged.

10.4.3.1.2.1 The data may be usable if the response for the verification exceed high (high bias) and the associated samples are non-detects.

10.4.3.1.2.2 If the criteria for the CCV is low (low bias), those sample results may be reported only if they exceed a maximum regulatory limit/decision level.

10.4.3.2 If the calibration verification is being performed using an auto sampler for night batch, two (2) vials of standard solution are placed in the device for analysis. The second standard must meet continuing calibration criteria and is used for calibration verification. The second check may be discarded only if there is a purge failure or incorrect spike concentration provided the first calibration standard meets the requirement. In this case, the first calibration standard is used as calibration verification following team leader/manager approval. Document this occurrence on instrument log.

10.4.3.2.1 Both CCVs must be evaluated. If vial 1 fails and vial 2 passes this meets the criteria of 10.4.3 of consecutive and immediate passing CCV.

10.4.3.2.2 If CCV number 2 fails, the analysis cannot continue unless it was determined that there was an isolated mechanical failure.

10.4.4 If any of the internal standard areas change by a factor of two (- 50% to + 100%) or the retention time changes by more than 30 seconds from the midpoint standard of the last initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate.

10.4.4.1 Reanalyze the continuing calibration standard. New initial calibration is required if reanalyzed standard continues to fail the internal standard requirements.

10.4.4.2 All samples analyzed while the system was out of control must be reanalyzed following corrective action.

10.5 Corrective Action Maintenance for Failed Tuning and Calibration Procedures

10.5.1 Inability to achieve criteria for instrument tuning or calibration may indicate the need for instrument maintenance. Maintenance may include routine system cleaning and replacement of worn expendables or the need for outside service if the scope of the repair exceeds the capability of the staff.

10.5.2 If maintenance is performed on an instrument, return to control must be demonstrated before analysis can continue. Return to control is demonstrated as follows:

10.5.2.1 Successful instrument tune using PFTBA.

10.5.2.2 Successful tune verification by the analysis of 4-bromofluorobenzene.

10.5.2.3 Successful initial calibration or continuing calibration.

11.0 PROCEDURE

11.1 Instrument conditions.

11.1.1 Recommended instrument conditions are listed in Table 2 and 2a (SIM only). Modifications of parameters specified with an asterisk are allowed as long as criteria of calibration are met. Any modification must be approved by team leader/manger.

11.1.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, use the same GC conditions for the analysis of all standards, blanks, samples, and QC samples.

11.2 Purge and Trap Device conditions.

11.2.1 See Table 2.

11.2.2 Daily Maintenance. Routine Daily maintenance must be performed before any tuning, calibration or sample analysis activities are initiated. These include checks of the following items:

<u>Purge and Trap Device:</u>

- Clean & bake purge tube.
- Bake trap and transfer lines.
- Check or refill internal/surrogate spike solution on SIM/SAM vials.
- Clean/replace syringe (if necessary).
- Change and refill rinse bottle.
- Empty and rinse waste bottle.

11.3 Step 1: GC/MS performance check.

- Inject 50ng of BFB solution directly on column or
- Purge 50ng/mL of BFB solution onto the GC column.

11.3.1 The GC/MS system must be checked to verify acceptable performance criteria are achieved (see Table 3).

11.3.2 **This performance test must be passed before each initial calibration.** Evaluate the tune spectrum by selecting the mass spectrum at the peak apex or by using an average spectrum (e.g., three highest abundance spectra, or across entire BFB peak).

11.3.2.1 Select the scans at the peak apex and one to each side of the apex.

11.3.2.2 Calculate an average of the mass abundances from the three scans.

11.3.2.3 Background subtraction is allowed. Select a single mass spectrum or an average mass spectrum across a short time range acquired within 20 seconds of the elution of BFB. The background subtraction must be used only to eliminate column bleed or instrument background ions. Do not subtract part of the tuning compound peak.

11.3.3 If all the criteria are not achieved, the analyst must retune the mass spectrometer with team leader/manager and repeat the test until all criteria are met.

11.3.3.1 Alternatively, an additional scan on each side of the peak apex may be selected and included in the averaging of the mass. This will provide a spectrum of five averaged scans centered on the peak apex. NOTE: The selection of additional mass scans for tuning may only be performed with supervisory approval on a case-by-case basis.

11.3.3.2 Note: All subsequent standards, samples, MS/MSDs, BS, and blanks associated with a BFB analysis must use identical mass spectrometer conditions.

11.4 Step 2: Daily calibration check

11.4.1 Initial calibration

11.4.1.1 Refer to Section 10.2.

11.4.1.2 An initial calibration must be established (or reestablished) on each instrument:

- Prior to any sample analyses.

- Whenever a new column is installed;
- Whenever instrument adjustments that affect sensitivity are made; and
- Whenever a continuing calibration standard fails to meet the specified acceptance criteria, on the second trial.

11.4.2 Initial Calibration Verification - Second Source Calibration Check Standard

11.4.2.1 This standard is only analyzed when initial calibration provided. Refer to Section 10.3.

11.4.3 Continuing Calibration verification standard

11.4.3.1 Refer to Section 10.4.

11.4.4 The method blank (step 3) cannot be analyzed until the continuing calibration verification meets the criteria.

11.5 Step 3: Method blank

11.5.1 The acceptable method blank must be analyzed for every 12-hour time period or sooner.

11.5.1.1 Water and medium-level soil samples - Place a 40 ml vial, filled with DI water onto the autosampler.

11.5.1.2 Low-level soil samples without sodium bisulfate - Transfer 5 ml of DI water to a 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray.

11.5.1.2.1 Low-level soil samples with sodium bisulfate (Method 5035) - Add 1g of sodium bisulfate into a 40 ml vial before adding 5 ml of DI water. Cap the vial with a Teflon septum, then place the vial onto the autosampler.

11.5.2 Program the autosampler to add internal standard and surrogate solution to the method blank for a concentration of 50 µg/l for each internal standard and surrogate.

11.5.2.1 For O.I. SIM spiker: Automatically adds 10 µl of 25 µg/ml internal standard and surrogate solution (Section 9.4.1.1) to the method blank.

11.5.2.2 For O.I. SAM spiker: Automatically adds 1 µl of 250 µg/ml internal standard and surrogate solution (Section 9.4.1.2) to the method blank.

11.5.3 No compound can be present above the laboratory's MDL. Common laboratory solvents (i.e. methylene chloride, acetone, hexane) may be present up until the RL. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e., targets are not present in samples or sample concentrations are $\geq$10x the blank).

11.5.4 Surrogates must meet recovery criteria specified in house limits.

11.5.5 If the method blank does not meet surrogate criteria or contains target analytes above the MDL, then

11.5.5.1 All samples analyzed following an out of control method blank must be reanalyzed if conditions in 11.5.3 are not met.

11.5.5.2 Check for the potential of contamination interference from the following areas. Make sure all items are free contamination.

- the analytical system,
- dust and vapor in the air,
- glassware and
- Reagents.

11.5.5.3 Re-analyze the method blank following the system evaluation. In this situation, the instrument logbook must have clearly documented notations as to what the problem was and what corrective action was implemented to enable the second blank to pass.

11.5.5.4 If re-analyzed method blank remains out of control, notify team leader or manager.

11.5.6 If two consecutive method blanks are analyzed during unattended operations, the second analysis must meet criteria for the subsequent sample analysis to be valid. Always report the second method blank. The second analysis can only be discarded because of a purge failure provided that the first blank meets the requirement. In this case, the first blank is reported following team leader/manager approval. Document this occurrence on the instrument log.

11.5.7 Re-analysis is not necessary if the analyte concentration falls well below the action or regulatory limit or if the analyte is deemed not important for the project.

11.6 Step 4: Blank spike (BS)

11.6.1 An acceptable blank spike must be analyzed with every analytical batch. The maximum number of samples per analytical batch is twenty.

11.6.2 Spike 50 ml of reagent water with appropriate amount of the standards to prepare a blank spike containing 50 μg/L of each analyte. In situations where lower detection limits are required, a blank spike at 20 μg/L may be prepared. The stock solution for the BS must be from the same source as the initial calibration solution. Refer to Table 8-F for the preparations of the blank spikes.

11.6.2.1 Water and medium-level soil samples - Place a 40 ml vial, filled with DI water onto the autosampler.

11.6.2.2 Low-level soil samples without sodium bisulfate - Aliquot 5 ml of the blank spike into vial and cap with Teflon septum, then place the vial into O.I. sample tray.

11.6.2.2.1 Low-level soil samples with sodium bisulfate for Method 5035 - Add 1g of sodium bisulfate to labeled 40 ml vial before aliquot 5 ml of the blank

spike into vial and cap with Teflon septum, then place the vial into O.I. sample tray.

11.6.3 Initiate auto addition of internal standard and surrogate into the syringe per 11.5.2.

11.6.4 Compare the percent recoveries (% R) (see Section 13.5) to the in-house limits acceptance criteria. If a blank spike is out of control, all the associated samples must be reanalyzed. The exception is if the blank spike recovery is high and no hits reported in associated samples and QC batch. In that case, the sample results can be reported with footnote (remark) and no further action is required. Or if the blank spike recovery is low and the hits in the samples are above regulatory levels.

11.6.5 Do not analyze samples and MS/MSD (step 5) unless the BS meets acceptance criteria.

11.6.6 The blank spike and matrix spike must be the same source and concentration.

11.7 Step 5: Samples /MS/MSD analysis

11.7.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

11.7.2 Select the sample dilution factor to assure the highest concentration analyte is above the calibration range midpoint, but below the upper limit of the range depend on project requirements. See Table 9 for dilution guideline.

- Utilize FID screen data.
- Utilize acquired sample data.
- Utilize the history program.
- Sample characteristics (appearance, odor).

11.7.3 Water samples.

11.7.3.1 Using O.I. Model 4560 sample concentrator with 4551 or 4552 vial multisampler,

- Place the 40 ml vial in the tray, or
- Load 5ml sample into purge tube if sample volume limited.

11.7.3.2 A matrix spike and matrix spike duplicate are performed independently by spiking 20ul of the appropriate standards into the 40ml sample vial. If there are not enough vials for this procedure, a matrix spike and a sample duplicate are performed in place of an MS/MSD.

11.7.4 Sediment/ soil sample

11.7.4.1 Low-level soil method

11.7.4.1.1 Collect the sample using the procedures detailed in the SOP for SW846 Method 5035 low - level soil samples.

11.7.4.1.2 Weigh out 5 g of each sample into a labeled, tared vial filled with 5 ml DI water. Add the matrix spike by manually puncturing the septum with a

small-gauge needle. Transfer the 40ml vial to the autosampler tray. Stir and heat the sample at the time of analysis.

11.7.4.2 Medium-level soil method

11.7.4.2.1 Collect the sample using the procedures detailed in the SOP for SW846 Method 5035 medium - level soil samples.

11.7.4.2.2 Select a methanol aliquot of appropriate volume (see Table 9) determined via screening and transfer to 40 ml of reagent water.

11.7.5 Program the autosampler to inject the internal standard and surrogate solution into the robotic syringe used to withdraw sample from the 40 ml vial. This addition to 5 ml of sample is equivalent to a concentration of 50 µg/L of each internal standard and surrogate.

11.7.5.1 For O.I. SIM spiker: Automatically adds 5 µl of 50 µg/ml internal standard and surrogate solution (Section 9.4.1.1) to each sample.

11.7.5.2 For O.I. SAM spiker: Automatically adds 1 µl of 250 µg/ml internal standard and surrogate solution (Section 9.4.1.2) to each sample.

11.7.6 Purge the sample for 9 minutes with Helium or Nitrogen.

11.7.6.1 Low-level soil sample must be performed at 40 °C while the sample is being agitated with the magnetic stirring bar or other mechanical means.

11.7.6.2 To improve the purging efficiency of water-soluble compounds, aqueous samples may also be purged at 40 °C as long as all calibration standards (for 1,4-Dioxane SIM option, purge temperature is 80°C), samples and QC samples are purged at the same temperature and acceptable method performance is demonstrated.

11.7.7 One sample is randomly selected from each analytical batch of similar matrix types and spiked in duplicate to determine whether the sample matrix contributes bias to the analytical results. A matrix spike and matrix spike duplicate are performed by spiking the sample for a concentration of 50 µg/l or 50 µg/kg based on 5 g dry weight. In situations where lower detection limits are required, a blank spike at lower concentration may be prepared.

11.7.8 Desorb the sample for a maximum of 4 minutes by rapidly heating the trap to 190 °C while backflushing with Helium. Desorb time may require performance optimization between 0.5 and 4.0 minutes as dictated by trap manufacturers specifications or instrument characteristics.

11.7.9 Program the purge and trap system to automatically rinse purge tube at least twice with heated organic-free water (reagent water) between analyses to avoid carryover of target compounds. For samples containing large amounts of water-soluble materials, suspended solids, high-boiling compounds, or high purgeable levels, it may be necessary to wash out the purging device with methanol solution between analyses, rinse it with distilled water.

11.7.10 Bake the trap at least 10 minutes at 210 °C to remove any residual purgeable compounds.

11.7.11 If the initial analysis of the sample or a dilution of the sample has a response for any ion of interest that exceeds the working range of the GC/MS system, the sample must be reanalyzed at a higher dilution.

11.7.11.1 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

11.8 Sample dilutions

11.8.1 Using Screening Data to Determine Dilution Factors

11.8.1.1 Dilution for High Concentration Analytes Exceeding the Calibration Range

11.8.1.1.1 The highest concentration target compound detected in the screen data is compared to the highest concentration calibration standard used for determinative volatile organics analysis.

11.8.1.1.1.1 Divide the calibration concentration of the screen concentration by the highest concentration calibration standard.

11.8.1.1.1.2 If the result is >1, sample dilution is considered.

11.8.1.1.2 The result from step 11.8.1.1.1 determines the dilution factor. The dilution factor is targeted to assure that the highest concentration diluted analyte is at the mid-range concentration of the calibration curve for the determinative analysis.

11.8.1.1.3 In all cases a conservative approach to dilution is applied to minimize the increase of detection and reporting limits

11.8.1.2 Dilution for High Concentration Matrix Interferences

11.8.1.2.1 The peak height of the background is compared to the peak height of the later eluting calibration standards from the screening analysis.

11.8.1.2.1.1 A rough estimate of background concentration is calculated by dividing the background peak height by the peak height of the selected screening standard and multiplying by its concentration.

11.8.1.2.2 If the result is >1, sample dilution is considered.

11.8.1.2.3 The result from step 11.8.1.2.1 determines the dilution factor. The dilution factor is targeted to avoid Carry-over contamination between

samples and facilitate qualitative and quantitative analysis of target compounds present in the sample.

11.8.1.2.4 In all cases a conservative approach to dilution is applied to minimize the increase of detection and reporting limits

11.8.2 If the concentration of any target compound in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and re-analyzed. Until the diluted sample is in a sealed sample vial, all steps in the dilution procedure must be performed without delay.

11.8.3 Water Samples.

11.8.3.1 Prepare all dilutions of water samples in volumetric flasks or Class A graduated cylinder. Intermediate dilutions may be necessary for extremely large dilutions.

11.8.3.2 Calculate the approximate volume of reagent water, which will be added to the volumetric flask or graduated cylinder and add slightly less than this quantity to the flask. Refer to Table 9 for dilution guideline.

11.8.3.3 Inject the proper sample aliquot from a syringe into the volumetric flask or graduated cylinder. It is also permissible to pour the sample directly into a graduated cylinder for some dilutions. Dilute the flask to the volume mark with reagent water. Cap the flask and invert the flask three times.

11.8.3.4 Fill a 40 ml sample vial and seal with a Teflon baked silicon septum, load the diluted sample into the autosampler and analyze according to Section 11.7.

11.8.4 Low-level Soil Samples.

11.8.4.1 Screen data is used to determine the appropriate sample preparation procedure for a sample, the low-level soil method or the medium-level soil method.

11.8.4.2 If any target compound exceeds the initial calibration range from the analysis of 5 g sample, a smaller sample size must be analyzed. However, the smallest sample size permitted is 0.5 g. If smaller than 0.5 g sample size is needed to prevent any target compounds from exceeding the initial calibration range, the medium level method must be used.

11.8.4.2.1 Samples that must use medium level method will be analyzed under separate calibration curve.

11.9 Data interpretation

11.9.1 Qualitative identification.

11.9.1.1 The targeted compounds shall be identified by analyst with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound.

11.9.1.2 The characteristic ions for target compounds that can be determined are listed in Table 7. Table 4 and Table 5 list the characteristic ions for internal standards and surrogate compounds respectively.

11.9.1.3 The criteria required for a positive identification are listed below.

11.9.1.3.1 The sample component must elute at the same retention time (RT) as the daily standard. Criteria are the RT of sample component must be within ± 10 seconds of the standard component (delta RT 0.17 minute) or within ±10 seconds relative to the shift of the associated Internal standard (delta RT of the IS ± 0 seconds).

11.9.1.3.2 The relative intensities of these ions must agree within ± 30 % between the daily standard and sample spectra. (Example: For an ion with an abundance of 50 % in the standard spectra, the corresponding sample abundance must be between 20 and 80 %.)

11.9.1.3.2.1 Compounds can have secondary ions outside criteria from co-eluting compounds and/or matrix effect that can contribute to ion abundances. The interference on ion ratios can't always be subtracted out by software programs resulting in qualified compound identification.

11.9.1.3.2.2 Quantitation reports display compounds that have secondary ions outside the ratio criteria with a "#" flag.

11.9.1.3.3 Structural isomers that produce very similar mass spectra must be identified as individual isomers if they have sufficiently different GC retention times. Isomers are considered resolved if the height of the valley between two isomer peaks is less than 50 % of the average of the two peak heights or 1- [valley height]/ [average peak height] is ≥50%. Otherwise, structural isomers are identified as isomeric pairs. The resolution of structural isomers must be verified in the ICV and daily CCV standards to verify if the instrument performance is adequate regarding separation of compound of interest which are structural isomers.

11.9.2 Quantitative analysis

11.9.2.1 Once a target compound has been identified, its concentration (Section 13.4) will be based on the integrated area of the quantitation ion, normally the base peak (Table 7). The compound is quantitated by internal standard technique with an average response factor generated from the initial calibration curve.

11.9.2.2 If the sample produces interference for the primary ion, use a secondary ion to quantitate (see Table 7). This is characterized by an excessive background signal of the same ion, which distorts the peak shape beyond a definitive integration. Also, interference could severely inhibit the response of the internal standard ion. This secondary ion must also be used to generate new calibration response factors.

11.10 Library search for tentatively identified compounds.

11.10.1 If a library search is requested, the analyst must perform a forward library search of NBS or NIST08 mass spectral library to tentatively identify 15 non-reported compounds.

11.10.2 Guidelines for making tentative identification are listed below.

11.10.2.1 These compounds must have a response greater than 10 % of the nearest internal standard. The response is obtained from the integration for peak area of the Total Ion Chromatogram (TIC).

11.10.2.2 The search is to include a spectral printout of the 3 best library matches for a particular substance. The results are to be interpreted by analyst.

11.10.2.3 Molecular ions present in the reference spectrum must be present in the sample spectrum.

11.10.2.4 Relative intensities of major ions in the reference spectrum (ions > 10 % of the most abundant ion) must be present in the sample spectrum.

11.10.2.5 The relative intensities of the major ions must agree within ± 20 %. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must between 30 and 70%).

11.10.2.6 Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.

11.10.2.7 Ions present in the reference spectrum but not in the sample spectrum must be verified by performing further manual background subtraction to eliminate the interference created by coeluting peaks and/or matrix interference.

11.10.2.8 Quantitation of the tentatively identified compounds is obtained from the total ion chromatogram based on a response factor of 1 and is to be tabulated on the library search summary data sheet.

11.10.2.9 The resulting concentration must be reported indicating: (1) that the value is estimate, and (2) which internal standard was used to determine concentration. Quantitation is performed on the nearest internal standard.

11.10.2.10 Mass spectral library search algorithm typically assign a match factor to the peak identity based on comparison of an unknown mass spectrum to library spectra. For spectra meeting the above conditions, match factors greater than 80% may be considered confirming evidence.

11.11 An instrument blank is a system evaluation sample containing lab reagent grade water with internal standards and surrogates. An instrument blank is used to remove and or evaluate residual carryover from high level standards, spike samples and field samples. Since target

compound lists have expanded to overlap some volatile and semi-volatile compounds, instrument blanks are necessary to remove carryover contamination.

11.11.1 The compounds that may exhibit carryover for this method are listed in Table 11.

11.11.2 If instrument blanks following a standard or spike sample exhibits carry-over effect, then any samples that show the same carryover profile, after a comparable concentration must be considered suspect and rerun for confirmation. For example, if an instrument blank has 1ppb detected after a 200ppb standard, then any sample following a sample containing 200ppb or above of the same compound must be confirmed for possible carryover.

11.11.3 If an Instrument Blank(s) was run following suspect high concentration samples and it exhibits the same carryover profile after a comparable concentration must be considered suspect and rerun for confirmation.

11.11.4 In some cases, several instrument blanks may have to be run to eliminate contamination from over loaded samples.

11.11.5 The analytical system is considered free of carryover, when no target analytes can be detected above the MDL.

11.12 Selected Ion Monitoring (SIM) Option

11.12.1 Instrument Set-Up: Modify the method for SIM analysis and define ion groups with retention times, ions and dwell times to include base peak ion for the target compounds of interest, surrogates, and internal standards (Table 2a.) Select a mass dwell time of 50 milliseconds for all compounds.

11.12.2 Calibration: Calibrate the mass spectrometer in the selected ion monitoring mode using 9 calibration standards of 0.25, 0.4, 1, 2, 5, 20, 50, 100 and 200 ug/l. Spike each standard with the SIM specific internal standard solution at 5 ug/ml. Calculate individual response factors and response factor RSDs. The initial calibration must meet the criteria in section 10.2.10.

11.12.3 Initial Calibration Verification. Verify the initial calibration after its completion using a 20 ug/l calibration standard purchased or prepared from a second standards reference materials source. The initial calibration verification must meet the criteria from Section 10.3.

11.12.4 Continuing Calibration Verification. Verify the initial calibration every 12 hours using a 5 or 20 ug/l calibration. The continuing calibration verification must meet the criteria from Section 10.4.

11.12.5 Surrogate Standard Calculation. Report surrogate spike accuracy for the surrogates spiked for the full scan GC/MS analysis.

12.0 QUALITY CONTROL

12.1 QC Requirements Summary

BFB	Before every initial calibration
Initial Calibration	Whenever needed
ICV - Second Source Calibration Check Standard	Following initial calibration
Calibration Verification Standard	Every 12 hours
Method Blank	Every 12 hours
Blank Spike	One per analytical batch*
Matrix Spike	One per analytical batch*
Matrix Spike Duplicate or Sample DUP (depends on sample volume)	One per analytical batch*
Surrogate	Every sample and standard
Internal Standard	Every sample and standard

*The maximum number of samples per analytical batch is twenty.

12.2 GC/MS Performance Check - BFB

12.2.1 Refer to Section 11.3.

12.3 Initial calibration

12.3.1 Refer to Section 10.2

12.4 Second Source Calibration Check Standard

12.4.1 Refer to Section 10.3.

12.4.2 Calibration Verification Standard

12.4.3 Refer to Section 10.4.

12.4 Method Blank

12.4.1 Refer to Section 11.5

12.5 Blank Spike

12.5.1 Refer to Section 11.6

12.6 Matrix Spike (MS)/Matrix Spike Duplicate (MSD)

12.6.1 One sample is selected at random from each analytical batch of similar matrix types and spiked in duplicate to check precision and accuracy.

12.6.2 Assess the matrix spike recoveries (Section 13.5) and relative percent difference (RPD) (Section 13.6) against the control limits.

12.6.3 If the matrix spike recoveries do not meet the criteria, check the blank spike recovery to verify that the method is in control. If the blank spike did not meet criteria, the method is out of control for the parameter in question and must be reanalyzed or qualified with an

estimate of potential bias. Otherwise, matrix interference is assumed and the data is reportable. No further corrective action is required.

12.7 Surrogates

12.7.1 All standards, blanks, samples, and matrix spikes contain surrogate compounds, which are used to monitor method performance. If the recovery of any surrogate compound does not meet the control limits, the result must be flagged and:

12.7.1.1 The calculation must be checked.

12.7.1.2 The sample must be reanalyzed if the recovery of any one surrogate is out of control limit.

12.7.2 If the sample exhibits matrix interference, defined as excessive signal levels from target or non-target interfering peaks. In this case, reanalysis may not be required following team leader/manager approval.

12.7.3 If surrogate recoveries are acceptable upon reanalysis, the data from the reanalysis is reported. If the reanalysis date did not meet the hold time, then both sets of data must be submitted with the reanalysis reported.

12.7.4 If surrogates are still outside control limits upon reanalysis, then both sets of data must be submitted with the first analysis reported.

12.8 Internal Standard

12.8.1 Retention time for all internal standards must be within ± 30 seconds of the corresponding internal standard in the latest continuing calibration or 50 μg/l standard of initial calibration

12.8.2 The area (Extracted Ion Current Profile) of the internal standard in all analyses must be within 50 to 200 % of the corresponding area in the latest calibration standard (12 hr. time period).

12.8.3 If area of internal standard does not meet control limits, the calculations must be checked. If a problem is not discovered, the sample must be reanalyzed.

12.8.4 If areas are acceptable upon reanalysis, the reanalysis data is reported.

12.8.5 If areas are unacceptable upon reanalysis, then both sets of data are submitted with the original analysis reported.

13.0 CALCULATION

13.1 Response Factor (RF)

$$RF = \frac{As \times Cis}{Ais \times Cs}$$

where:

As = Area of the characteristic ion for the compound being measured.
Ais = Area of the characteristic ion for the specific internal standard.
Cs = Concentration of the compound being measured (ug/l).
Cis = Concentration of the specific internal standard (ug/l).

13.2 Percent Relative Standard Deviation (% RSD)

$$\%RSD = \frac{SD}{RFav} \times 100$$

where:
SD = Standard Deviation
RFav = Average response factor from initial calibration.

13.3 Percent Difference (%D)

$$\%D = \frac{(RFav - RFcv)}{RFav} \times 100$$

where:
RFcv = Response factor from Calibration Verification standard.
RFav = Average response factor from initial calibration.

13.4 Concentration (Conc.)

For water:

$$\text{Conc. } (\mu g/l) = \frac{Ac \times Cis \times Vp}{Ais \times RF \times Vi}$$

For soil/sediment low level (on a dry weight basis):

$$\text{Conc. } (\mu g/kg) = \frac{Ac \times Cis \times Vp}{Ais \times RF \times Ws \times M}$$

For soil/ sediment medium level (on a dry weight basis)

$$\text{Conc. } (\mu g/kg) = \frac{Ac \times Cis \times Vp \times Vt}{Ais \times RF \times Vme \times Ws \times M}$$

Where:
Ac = Area of characteristic ion for compound being measured.
Ais = Area of characteristic ion for internal standard.
Cis = Concentration of internal standard
RF = Response factor of compound being measured(from initial calibration)
Vi = Initial volume of water purged (ml)
Vp = 5 ml (Total Purge Volume)
Vme = Volume of Methanol aliquot
Vt = Ml Solvent + ((100-% solid)/100 x Ws)

Ws = Weight of sample extracted (g).
M = (100 - % moisture in sample) / 100 or % solids / 100

13.5 Percent Recovery (% R)

$$\%\ R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

13.6 Relative Percent Difference (RPD)

$$RPD = \frac{|\ MSC - MSDC\ |}{(1/2)\ (MSC+MSDC)} \times 100$$

Where:
MSC = Matrix Spike Concentration
MSDC = Matrix Spike Duplicate Concentration

13.7 Linear regression by the internal standard technique.

$$C_s = \left(\frac{\frac{A_s}{A_{is}} - b}{a} \right) \times C_{is}$$

Where:
Cs = concentration of target analyte
As = Area of target analyte
Cis = concentration of the internal standard
b = Intercept
a = slope of the line

$$a = \frac{N \Sigma xy - \Sigma x \Sigma y}{N \Sigma x^2 - (\Sigma x)^2}$$

$$b = \frac{\Sigma y - a \Sigma x}{N}$$

N = number of points
x = amount of analyte
y = response of instrument

13.8 Correlation Coefficient

$$r = \frac{\Sigma(x - \bar{x})(y - \bar{y})}{\sqrt{\Sigma(x - \bar{x})^2 \Sigma(y - \bar{y})^2}}$$

Where r = correlation coefficient
x = amount of analyte

y = response of instrument

$\bar{x}$ = average of x values

$\bar{y}$ = average of y values

13.9 Quadratic curve with internal standard technique

$$Cs = \frac{-b \pm \sqrt{b^2 - 4a\left(c - \frac{A_s \times C_{is}}{A_{is}}\right)}}{2a}$$

Where:
Cs = concentration of target analyte
As = Area of target analyte
Cis = concentration of the internal standard
b = Intercept
a = slope of the line

14.0 DOCUMENTATION

14.1 The Analytical Logbook. The logbook must be completed by the analyst daily. Each instrument will have a separate logbook. The daily sequence must be recorded in the logbook by giving a file number to every instrument standard, QC, and samples in appropriate spaces. The files must be never overwritten or skipped intentionally. In case where the file is skipped or overwritten, a thorough explanation must be documented in the notes section. Upon completion, every analytical batch must be reviewed and signed by a supervisor/team lead. Supervisor signature indicates all documentation was performed correctly.

14.1.1 If samples or blank spike require reanalysis, a brief explanation of the reason and corrective action must be documented in the Comments section.

14.1.2 If maintenance was done on the instrument in order to pass the CCV or any other reason, the analyst must document it in the logbook.

14.2 Standards Preparation Logbook must be completed for all standard preparations. All information must be completed; the page must be signed and dated by the appropriate person.

14.2.1 The SGS lot number must be cross-referenced on the standard vial.

14.3 Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

14.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

14.5 Supervisory personnel must review and sign all laboratory logbooks monthly to ensure that information was recorded properly. Additionally, the instrument maintenance logbooks and the accuracy of the recorded information must also be verified and signed off on the first page of the logbook quarterly by a supervisor/team lead.

14.6 Acrolein and Acrylonitrile data reported from a preserved sample must be footnoted: “Results reported from the HCl preserved sample. This reported result can only be used for screening purposes for Acrolein and Acrylonitrile.” Any samples analyzed form an unpreserved vial must be footnoted stating samples were unpreserved and analyzed within 7 days.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes

15.2.2 Hazardous aqueous wastes

15.2.3 Chlorinated organic solvents

15.2.4 Non-chlorinated organic solvents

15.2.5 Hazardous solid wastes

15.2.6 Non-hazardous solid wastes

Table 1 TARGET COMPOUNDS		
Acetone	1,4-Dichlorobenzene	Methylene Bromide
Acetonitrile	Dichlorodifluoromethane	Methylene Chloride
Acrolein	1,1-Dichloroethane	1-Methylnaphthalene
Acrylonitrile	1,2-Dichloroethane	2-Methylnaphthalene
Allyl Chloride	1,1-Dichloroethene	Naphthalene
Benzene	cis-1,2-Dichloroethene	2-Nitropropane
Benzyl chloride	trans-1,2-Dichloroethene	Pentachloroethane
Bromobenzene	1,2-Dichloropropane	Propionitrile
Bromochloromethane	1,3-Dichloropropane	Propyl Acetate
Bromodichloromethane	2,2-Dichloropropane	n-Propylbenzene
Bromoform	1,1-Dichloropropene	Styrene
Bromomethane	cis-1,3-Dichloropropene	Tert Butyl Alcohol
2-Butanone (MEK)	trans-1,3-Dichloropropene	tert-Amyl Methyl Ether
Butyl Acetate	1,4-Dioxane	tert-Butyl Ethyl Ether
n-Butyl Alcohol	Epichlorohydrin	1,1,1,2-Tetrachloroethane
n-Butylbenzene	Ethyl Acetate	1,1,2,2-Tetrachloroethane
sec-Butylbenzene	Ethyl Ether	Tetrachloroethene
tert-Butylbenzene	Ethyl Methacrylate	Tetrahydrofuran
Carbon Disulfide	Ethylbenzene	Toluene
Carbon Tetrachloride	p-Ethyltoluene	trans-1,4-Dichloro-2-Butene
Chlorobenzene	Freon 113	1,2,3-Trichlorobenzene
Chlorodifluoromethane	Heptane	1,2,4-Trichlorobenzene
Chloroethane	Hexachlorobutadine	1,1,1-Trichloroethane
2-Chloroethyl Vinyl Ether	Hexachloroethane	1,1,2-Trichloroethane
Chloroform	Hexane	Trichloroethene
Chloromethane	2-Hexanone	Trichlorofluoromethane
Chloroprene (2-chloro-1,3-butadiene)	Iodomethane (Methy iodide)	1,2,3-Trichloropropane
o-Chlorotoluene	IsoAmyl Alcohol	1,2,4-Trimethylbenzene
p-Chlorotoluene	Isobutyl Alcohol	1,3,5-Trimethylbenzene
Cyclohexane	Isopropyl Acetate	2,2,4 Trimethylpentane
Cyclohexanone	Isopropylbenzene	Vinyl Acetate
di-Isobutylene	p-Isopropyltoluene	Vinyl Chloride
di-Isopropyl Ether	Methacrylonitrile	Vinyltoluene
1,2-Dibromo-3-Chloropropane	Methyl Acetate	m,p-Xylene
Dibromochloromethane	3 Methyl-1-Butanol	o-Xylene
1,2-Dibromoethane	Methyl Tert Butyl Ether	Ethanol
Dibromomethane	Methylcyclohexane	Methyl Acrylate
1,2-Dichlorobenzene	Methyl Methacrylate	1-chloro-1,1-difluoroethane
1,3-Dichlorobenzene	4-Methyl-2-pentanone (MIBK)	1,1,1-trifluoroethane
1,1-dichloro-1-fluroethane	2,2-Dichloropropane	1,3-Butadiene
3,3-Dimethyl-1-Butanol	Tert-Butyl Formate	Tert-amyl alcohol
2-methylnaphthalene	1,2,3-Trimethylbenzene	

Table 2 RECOMMENDED OPERATING CONDITION	
Gas Chromatograph/ Mass Spectrometer	
Carrier Gas (linear velocity)	Helium at *30 cm/sec (or hydrogen)
Mass range	35 – 270 amu *
Electron Energy	70 volts (nominal)
Scan time	not to exceed 2 sec. per scan
Injection port temperature	200 - 225 °C
Source temperature	200 - 250 °C
Transfer line temperature	220 - 280 °C
Analyzer temperature	220 - 250 °C
Gas Chromatograph temperature program*	
Initial temperature	*40 °C
Time 1	*3 minutes
Column temperature rate	*8 degrees/min.
Final temperature	*220 °C.- 240 °C
Total run time	*25 – 50 mins
Split ratio	*20:1
Purge and Trap Device	
Purge time	9 min. (at 40 °C for low-level soil) SIM – 6 min @ 80 °C
Purge gas	Helium or Nitrogen
Desorb**	1 min. at 190 °C
Bake	>10 min. at 210 °C
Transfer line	100 - 130 °C
Valve temperature	approx. transfer line temperature

(*) Parameter modification allowed for performance optimization provided operational and QC criteria is achieved. (must be approved by team leader/manager)

(**) Desorb time may require performance optimum between 0.5 and 4.0 minutes as dictated by trap manufacturers specifications or instrument characteristics

Table 2a SIM Group Parameters		
Group No.	**Retention Time (minutes)**	**Ions**
1	0 – 10.8	58, 65, 66, 88
2	10.8 – 16.0	95, 174, 176, 96,64

Table 3 BFB KEY IONS AND ION ABUNDANCE CRITERIA	
Mass	**Ion Abundance Criteria**
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5-9% of mass 174
176	>95% and <101% of mass 174
177	5-9% of mass 176

Table 4 INTERNAL STANDARD QUANTITION IONS	
Internal Standard	**Primary/Secondary Ions**
1,4-Difluorobenzene	114 / 63,88
Chlorobenzene-d5	117 / 82, 119
Pentafluorobenzene	168
1,4-Dichlorobenzene-d4	152 / 115, 150
Tert Butyl Alcohol-d9	65/66
Internal Standard (SIM)	
4-BFB	95/174,176

Table 5 SURROGATE QUANTITION IONS	
Surrogate Compound	**Primary/Secondary Ions**
1,2 Dichloroethane – d_4	102
Dibromofluoromethane	113
Toluene-d8	98
4-Bromofluorobenzene	95 / 174, 176
1,4-dioxane-d8	96, 64

Table 7 Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)	Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)
Tert Butyl Alcohol-d9	65		Dibromomethane	93	95, 174
Tert Butyl alcohol	59	57	Di-isobutylene	57	
Ethanol	45	46	Epichlorohydrin (pp)	57	57, 49, 62, 51
1,4-Dioxane (pp)	88	58,43,57	Heptane	57	
Pentafluorobenzene	168		Methyl cyclohexane	83	
1,1,1-Trichloroethane	97	99, 61	Methyl methacrylate	100	69, 41, 39
1,1-Dichlorethane	63	65, 83	n-Butanol (pp)	56	41
1,1-Dichloroethene	96	61, 63	Propyl Acetate	43	
2,2-Dichloropropane	77	97	tert Amyl Methyl Ether	73	
2-Butanone (pp)	72	43, 72	Trichloroethene	95	97, 130, 132
Acetone (pp)	58	43	**Chlorobenzene-d5**	117	82,119
Acetonitrile (pp)	41	41, 40, 39	1,1,1,2-Tetrachloroethane	131	133, 119
Acrolein (pp)	56	55,58	1,3-Dichloropropane	76	78
Acrylonitrile (pp)	53	52, 51	Bromoform	173	175, 254
Allyl Chloride	76	41	Butyl Acetate	56	
Bromochloromethane	128	49, 130	Chlorobenzene	112	77, 114
Bromomethane	94	96	Dibromochloromethane	129	127
Carbon disulfide	76	78	Ethylbenzene	91	106
Carbon tetrachloride	117	119	m-Xylene	106	91
Chlorodifluouromethane	51	86	o-Xylene	91	106
Chloroethane	64	66	3,3-Dimethyl-1-Butanol	57	69
Chloroform	83	85	p-Xylene	106	91
Chloromethane	50	52	Styrene	104	78
Chloroprene	53	53, 88, 90, 51	Ethyl methacrylate	69	69, 41, 99, 86, 114
cis-1,2-Dichloroethene	96	61, 98	Toluene	92	91
Cyclohexane	84		**Toluene-d_8 (S)**	98	
Dibromofluoromethane (S)	113		Tetrachloroethene	164	129,131,166
Dichlorodifluoromethane	85	87	Cyclohexanone	55	
1,1-Dichloropropene	75	110, 77	2-Hexanone (pp)	58	43, 57, 100
Diethyl ether	74	45, 59	trans-1,3-Dichloropropene	75	77, 39
1,3-Butadiene	54		**1,4 Dichlorobenzene-d4**	152	115,150
Diisopropyl ether	45	102	1,1,2,2-Tetrachloroethane	83	131, 85
Ethyl acetate (pp)	45	43, 88, 61	1,2,3-Trichlorobenzene	180	182, 145
Ethyl tert Butyl Ether	59		1,2,3-Trichloropropane	110	77,75
Hexane	56		1,2,4-Trichlorobenzene	180	182, 145
Isopropyl acetate	87	43	1,2,4-Trimethylbenzene	105	120

Table 7 Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)	Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)
Tert-Amyl alcohol	59	73,55	1,2-Dibromo-3-chloropropane(pp)	157	155, 75
Freon 113	151		1,2-Dichlorobenzene	146	111,148
Iodomethane	142	127, 141	1,3,5-Trimethylbenzene	105	120
Isobutyl alcohol (pp)	43	43, 41, 42, 74	1,3-Dichlorobenzene	146	111, 148
Methacrylonitrile (pp)	67	41, 39, 52, 66	1,4-Dichlorobenzene	146	111, 148
Methyl Acetate	43	74	2-Chlorotoluene	126	91
Methylene chloride	84	86, 49	**4-Bromofluorobenzene (S)**	95	174, 176
Methyl-t-butyl ether	73	57	2-methylnaphthalene	142	141,115,143
Propionitrile (ethyl cyanide)(pp)	54	54, 52, 55, 40	Dibromofluoromethane		
Tetrahydrofuran	71	42	4-Chlorotoluene	91	126
trans-1,2-Dichloroethene	96	61, 98	Benzyl chloride	91	91, 126, 65, 128
Trichlorofluoromethane	101	151, 153	Bromobenzene	156	77, 158
Vinyl acetate	86	43	Hexachlorobutadiene	225	223, 227
Vinyl chloride	62	64	Hexachloroethane (pp)	201	166, 199, 203
Methyl Acrylate	85	55	Isopropylbenzene	105	120
Tert-Butyl Formate	59	57, 41	Naphthalene	128	-
1-chloro-1, 1-difluoroethane	65	45,85	n-Butylbenzene	92	91, 134
1,1,1-trifluoroethane	69	69,45	n-Propylbenzene	91	120
1,1-dichloro-1-fluroethane	81	45,61	Pentachloroethane (pp)	167	167,130,132,165,169
2,2-Dichloropropane	77	97,79	p-isopropyltoluene	119	134,91
1,2,3-Trimethyl benzene	105	120,119			
1,4 Difluorobenzene	114	63, 88	sec-Butylbenzene	105	134
1,1,2-Trichloroethane	83	97, 85	tert-Buytlbenzene	119	91, 134
1,2-Dibromoethane	107	109, 188	trans-1,4-Dichloro-2-butene (pp)	53	88, 75
1,2 Dichloroethane	62	98			
1,2 Dichloropropane	63	112	**(pp) = Poor Purging Efficiency**		
2,2,4 Trimethylpentane	57		(S)=Surrogate		
2-Chloroethyl-vinylether (pp)	63	65, 106			
Dichloroethane-d_4 (S)	65	102			
2-Nitropropane	46	-			
3 Methyl –1 butanol	70	55			
4-Methyl-2-pentanone (pp)	58	43, 85, 100			
Benzene	78	-			
Bromodichloromethane	83	85, 127			
cis-1,3-Dichloropropene	75	77, 39			
Methylcyclohexane	83				

Table 7-1 SIM - Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)
4-BFB	95	174, 176
1,4-Dioxane	88	58
1,4-dioxane-d8	96	64

Table 8 STANDARDS PREPARATION

A) Internal standard and Surrogate mixtures:

	a) 50/500 µg/ml	b) 250/2,500 µg/ml
Internal Standard Mixture (20,000 µg/ml)	0.25 ml	3.125 ml
Tert Butyl Alcohol-d_9 (50,000 µg/ml)	1.0 ml	12.5 ml
Surrogate Mixture (10,000 µg/ml)	0.5 ml	6.25 ml
Methanol	98.25 ml	228.125 ml
Total	100 ml	250 ml

- 50/500 µg/ml internal standard and surrogate mixture: The mixture is prepared by measuring 0.25 ml of 20,000 µg/ml Internal Standard Mixture (Ultra or equivalent), 1.0 ml of 50,000 µg/ml TBA-d_9 (Absolute or equivalent), 0.5 ml of 10,000 µg /ml Method 8260A Surrogate Standard Mixture (Absolute or equivalent) and bringing to 100 ml with methanol.

- 250/2,500 µg/ml internal standard and surrogate mixture: The mixture is prepared by measuring 3.125 ml of 20,000 µg/ml Internal Standard Mixture (Ultra or equivalent), 12.5 ml of 50,000 µg/ml TBA-d_9 (Absolute or equivalent), 6.25 ml of 10,000 µg /ml Method 8260A Surrogate Standard Mixture (Ultra or equivalent) and bringing to 250 ml with methanol.

B) Bromofluorobenzene (BFB):

	a) 25 µg/ml	b)) 250 µg/ml
BFB (25,000 µg/ml)	0.1 ml	0.1 ml
Methanol	99.9 ml	9.9 ml
Total	100 ml	10 ml

- 25 µg/ml solution for direct injection: The BFB is prepared at 25 µg /ml by measuring 0.1 ml of 25,000 µg /ml (Absolute Stock or equivalent) and diluting to 100 ml with methanol.

- 250 µg /ml solution for purging: The BFB is prepared at 250 µg /ml by measuring 0.1 ml of 25,000 µg /ml (Absolute Stock or equivalent) and diluting to 10 ml with methanol.

C) Secondary dilution standards:

2nd Dilution Standards	Stock Solution	Concentration (µg/ml)	Volume Added (µl)	Final Volume in Methanol (ml)	Final Concentration (µg/ml)
V8260 Mixture	502.2 CAL2000 Mega Mix	2,000	5,000	100	100
	524 Cal Mix 7A	2,000	5,000		100
	524 Cal Mix 8	2,000	5,000		100
	Ketone mix	20,000	1,500		300
	Multiple neat compounds	Neat			100
	Multiple single component standards	5000-20,000	500-2000		100
Acrolein	Acrolein	Neat (95%)	12.5	100 (4:1 ratio of DI water : methanol)	100

Gas Mixture	VOC Gas Mixture	2,000	1,250	25	100
	Cyclohexane	neat	3.2		100

- 100 µg /ml V8260 mixture: The mixture is prepared at 100 µg /ml by measuring 5 ml of 2,000 µg/ml 502.2 Mega Mix stock standard, 5 mL of 2,000 µg/ml 524 Cal Mix 7A, 5 mL of 2,000 µg/ml 524 Cal Mix 8, appropriate amount of some neat compounds and single component standards, and bringing to 100 ml with methanol.

- 100 µg/ml V8260 Acrolein: The mixture is prepared at 100 µg/ml by measuring 12.5 µl of 95% acrolein to 100 ml of a 4:1 ratio of DI water:methanol to reduce the amount of methanol injected into the system per run.

- 100 µg /ml gas mixture ***: The mixture is prepared at 100 µg /ml by measuring 1.25 ml of 2,000 µg/ml stock standard, 3.2 µl of neat cyclohexane and bring to 25 ml with methanol.
  ******* Gas mixture must be prepared weekly.

D).1 Initial Calibration Standards: using DI water bring to 50 ml final volume for the 1 -400 ppb standards and 500 ml for the 0.2 and 0.5 ppb standards: All mixtures used must be **secondary dilution** standards at **100 ppm**. Note: Larger volumes may be prepared if needed i.e. if 100 ml final volume is used the volume of the standard added would be doubled.

Standard and Surrogate Concentration	V8260 Mix (100 ppm)	V8260 Acrolein (100 ppm)	Gas compound Mix (100 ppm)	Surrogate Mix when added manually (100ppm)
0.2 ppb	1.0 µl	1.0 µl	1.0 µl	1.0 µl#
0. 5 ppb	2.5 µl	2.5 µl	2.5 µl	2.5 µl#
1 ppb	0.5 µl	0.5 µl	0.5 µl	0.5 µl#
2 ppb *	1.0 µl	1.0 µl	1.0 µl	1.0 µl#
4 ppb *	2.0 µl	2.0 µl	2.0 µl	2.0 µl#
5 ppb	2.5 µl	2.5 µl	2.5 µl	2.5 µl#
8 ppb *	4.0 µl	4.0 µl	4.0 µl	4.0 µl#
10 ppb *	5 µl	5 µl	5 µl	5 µl#
20 ppb	10 µl	10 µl	10 µl	10 µl#
50 ppb	25 µl	25 µl	25 µl	25 µl#
100 ppb	50 µl	50 µl	50 µl	50 µl#
200 ppb	100 µl	100 µl	100 µl	100 µl#
300 ppb *	150 µl	150 µl	150 µl	150 µl#
400 ppb *	200 µl	200 µl	200 µl	200 µl#

* depending upon the instrument.
\# See Section 10.2.2.1 for correction factor.

- When calibrating for Method 5035 low-level soil samples, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of each standard into vial if applicable. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds.

Table 8 STANDARD PREPARATION (Continued)

D).2 Initial Calibration Standards for 1,4-Dioxane using SIM

Standard Concentration (ppb)	1,4-Dioxane Solution (100ppm)	DI Water – Final Volume (ml)
0.25	1.25 µl	500
0.4	2 µl	500
1	1 µl	100
2	2 µl	100
5	2.5 µl	50
20	10 µl	50
50	25 µl	50
100	50 µl	50

E) C**ontinuing Calibration Standard: using DI water bring to 50 ml final volume:** All mixtures used are secondary dilution standards at 100 ppm.

Concentration	V8260 Mix (100 ppm)	V8260 Acrolein (100 ppm)	Gas compound Mix (100 ppm)
50 ppb	25 µl	25 µl	25 µl

- When calibrating for Method 5035 low-level soil samples, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of the continuing calibration standard into vial if applicable. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds.

F) Blank Spike (BS): using DI water bring to 50 ml final volume: All mixtures used are 100 ppm secondary dilution standards.

Concentration	V8260 Mix (100 ppm)	V8260 Acrolein (100 ppm)	Gas compound Mix (100 ppm)
50 ppb	25 ul	25 ul	25 ul

For lower detection level required (test code: V8260LL)

Concentration	V8260 Mix (100 ppm)	V8260 Acrolein (100 ppm)	Gas compound Mix (100 ppm)
20 ppb	10 ul	10 ul	10 ul

- When calibrating for Method 5035 low-level soil samples, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of the blank spike into vial if applicable. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds.

Table 9 GUIDELINE FOR DILUTION PREPARATION
Water Sample

Dilution	Sample amount taken	Final volume A (volumetric)	Take from final volume A	Final volume B (volumetric)
1:2	25 ml	50 ml		
1:5	10 ml	50 ml		
1:10	5 ml	50 ml		
1:20	2.5 ml	50 ml		
1: 25	2 ml	50 ml		
1:50	1 ml	50 ml		
1:100	0.5 ml	50 ml		
1:200	250 µl	50 ml		
1:250	200 µl	50 ml		
1:500	100 µl	50 ml		
1:1000	50 µl	50 ml		
1:2000	25 µl	50 ml		
1:2500	20 µl	50 ml		
1:5000	10 µl	50 ml		
1:10000	0.5 ml	50 ml	0.5 ml	50 ml
1:20000	0.5 ml	50 ml	250 µl	50 ml
1:25000	0.5 ml	50 ml	200 µl	50 ml
1:50000	0.5 ml	50 ml	100 µl	50 ml
1:100000	0.5 ml	50 ml	50 µl	50 ml

Soil-Low level (Non-Encore sample)

Dilution	Sample amount taken	Final volume
1:2	2.5 gram	5 ml
1:5	1 gram	5 ml
1:10	0.5 gram	5 ml

Soil-medium level

Additional Dilution	Sample in Methanol amount taken	Final volume (volumetric)
1:1	1 ml	50 ml
1:2	0.5 ml	50 ml
1:5	200 µl	50 ml
1:10	100 µl	50 ml
1:20	50 µl	50 ml
1: 25	40 µl	50 ml
1:50	20 µl	50 ml
1:100	10 µl	50 ml
1:200	5 µl	50 ml
1:250	4 µl	50 ml
1:500	2 µl	50 ml

Table 10 REPORTING LIMITS

Compound	Water	Soil	Compound	Water	Soil
	µg/l	µg/kg		µg/l	µg/kg
Chlorodifluoromethane	5	5	Chloroform	1	2
Dichlorodifluoromethane	2	5	Freon 113	5	5
Chloromethane	1	5	Methacrylonitrile	10	10
Vinyl chloride	1	2	Butyl Acetate	5	5
Bromomethane	2	5	1,1,1-Trichloroethane	1	2
Chloroethane	1	5	Heptane	5	5
Trichlorofluoromethane	2	5	n-Propyl acetate	5	5
Ethyl ether	2	2	2-Nitropropane	10	10
Acrolein	10	10	Tetrahydrofuran	10	10
1,1-Dichloroethene	1	1	2-Chloroethyl Vinyl Ether	10	25
Tertiary butyl alcohol	10	25	n-Butyl alcohol	250	250
Acetone	10	10	Cyclohexane	5	2
Methyl acetate	5	5	Carbon Tetrachloride	1	2
Allyl chloride	5	5	1,1-Dichloropropene	1	2
Acetonitrile	100	100	Isopropyl Acetate	5	5
Iodomethane	2	5	Benzene	0.5	0.5
Isobutyl alcohol	50	50	1,2-Dichloroethane	1	1
Carbon disulfide	2	2	Trichloroethene	1	1
Methylene chloride	2	5	Methyl methacrylate	10	10
Methyl tert butyl ether	1	1	1,2-Dichloropropane	1	1
Trans-1,2-Dichloroethene	1	1	2-ethylhexyl acrylate	2	2
Di-isopropyl ether	2	2	Dibromomethane	1	5
2-Butanone	10	10	1,4-Dioxane	130	130
1,1-Dichloroethane	1	1	Bromodichloromethane	1	2
Hexane	5	5	cis-1,3-Dichloropropene	1	2
Chloroprene	5	5	4-Methyl-2-pentanone	5	5
Acrylonitrile	10	10	Toluene	1	1
Vinyl acetate	10	10	trans-1,3-Dichloropropene	1	2
Ethyl acetate	5	5	Ethyl methacrylate	10	10
2,2-Dichloropropane	1	2	1,1,2-Trichloroethane	1	2
Cis-1,2-Dichloroethene	1	1	2-Hexanone	5	5
Bromochloromethane	1	5	Cyclohexanone	50	200

Table 10 REPORTING LIMITS (Continued)

Compound	Water	Soil	Compound	Water	Soil
	µg/l	µg/kg		µg/l	µg/kg
Tetrachloroethene	1	2	4-Chlorotoluene	2	2
1,3-Dichloropropane	1	2	1,3,5-Trimethylbenzene	2	2
Dibromochloromethane	1	2	tert-Butylbenzene	2	2
1,2-Dibromoethane	1	1	1,2,4-Trimethylbenzene	2	2
Chlorobenzene	1	2	sec-Butylbenzene	2	2
1,1,1,2-Tetrachloroethane	1	2	1,3-Dichlorobenzene	1	1
Ethylbenzene	1	1	p-Isopropyltoluene	2	2
m,p-Xylene	1	1	1,4-Dichlorobenzene	1	1
o-Xylene	1	1	1,2-Dichlorobenzene	1	1
Styrene	1	2	n-Butylbenzene	2	2
Bromoform	1	5	1,2-Dibromo-3-choropropane	2	2
Isopropylbenzene	1	2	1,2,4-Trichlorobenzene	1	5
Bromobenzene	1	5	Hexachlorobutadiene	2	5
1,1,2,2-Tetrachloroethane	1	2	Naphthalene	5	5
Trans-1,4-Dichloro-2-butene	5	5	1,2,3-Trichlorobenzene	1	5
1,2,3-Trichloropropane	2	5	Epichlorohydrin	100	100
n-Propylbenzene	2	2	3-Methyl-1-butanol	50	100
2-Chlorotoluene	2	2	Hexachloroethane	2	5
Ethanol	100	200	Methyl Acrylate	5	5
Benzyl Chloride	5	5	Methylcyclohexane	5	2
2,2,4-Trimethylpentane	5	5	1,1,1-trifluoroethane (Freon 143a)	5	5
1-chloro-1,1-difluoroethane (Freon 142b)	5	5	1,1-dichloro-1-fluoroethane (Freon 141b)	5	5
1,3-Butadiene	5	5	Pentachloroethane	5	5
1.4-Dioxane (SIM)	0.4	5	2-methylnaphthalene	5	5
Tert-Butyl Formate	5	5	Tert-amyl alcohol	25	25
1,2,3-Trimethylbenzene	2	2			

Table 11 COMPOUNDS THAT MAY EXHIBIT CARRYOVER

Compound
1,2,4-Trichlorobenzene
Hexachlorobutadiene
Naphthalene
1,2,3-Trichlorobenzene

Table 12 GUIDANCE MINIMUM RELATIVE RESPONSE FACTOR CRITERIA

Compound	Minimum Response Factor
Dichlorofluoromethane	0.01
Chloromethane	0.01
Vinyl chloride	0.01
Bromomethane	0.01
Chloroethane	0.01
Trichlorofuoromethane	0.01
1,1-Dichloroethene	0.06
Freon 113	0.05
Acetone	0.01
Carbon Disulfide	0.100
Methyl Acetate	0.01
Methylene chloride	0.01
trans-1,2 Dichloroethene	0.100
cis-1,2 Dichloroethene	0.2
Methyl tert-butyl Ether	0.100
1,1 Dichloroethane	0.3
2-Butanone	0.01
Chloroform	0.3
1,1,1 Trichloroethane	0.05
Cyclohexane	0.01
Carbon Tetrachloride	0.100
Benzene	0.2
1,2 Dichloroethane	0.07
Trichloroethene	0.200
Methylcyclohexane	0.05
1,2-Dichloropropane	0.2
Bromodichloromethane	0.3
cis-1,3-Dichloropropene	0.3
trans-1,3-dichloropropene	0.3

Table 12 cont'd Compound	Minimum Response Factor
Tetrachloroethene	0.1
2-Hexanone	0.01
Dibromochloromethane	0.2
1,2-Dibromoethane	0.2
Chlorobenzene	0.4
Ethyl benzene	0.4
m,p-Xylene	0.2
o-Xylene	0.2
Styrene	0.2
Bromoform	0.100
Isopropylbenzene	0.4
1,1,2,2-Tetrachloroethane	0.2
1,3-Dichlorobenzene	0.5
1,4-Dichlorobenzene	0.6
1,2-Dichlorobenzene	0.6
1,2-Dibromo-3-chloropropane	0.01
1,2,4-Trichlorobenzene	0.4
1,3-Butadiene	0.100
3,3-Dimethyl-1-butanol	0.010
1,4-Dioxane (SIM)	0.010
4-Methyl-2-Pentanone	0.03
Toluene	0.3
1,1,2-Trichloroethane	0.2
Bromochloromethane	0.1
1,2,3-Trichlorbenzene	0.4

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
10.4.1	Revised frequency of BFB
11.3.2	Revised frequency of BFB
12.1	Revised frequency of BFB

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
01	01/20/2021	Maria Ruschke
02	03/05/2021	Olga Azarian
03	07/22/2021	Olga Azarian
04	04/12/2022	Olga Azarian
05	04/25/2023	Olga Azarian

END OF DOCUMENT

LAB SUPERVISOR: _____

QA OFFICER: Olga L. Agosto

EFFECTIVE DATE: 9-21-2023

TITLE: METHOD 8270E, SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
REFERENCES: SW846 8270E, revision 6, June 2018
REVISED SECTIONS: 9.5.1.1, 10.1.1

1.0 SCOPE AND APPLICATION

1.1 The following method describes the analytical procedure that is utilized by SGS to analyze semi volatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and water samples. Options are incorporated for the analysis of sixteen (16) polyaromatic hydrocarbons (PAH) and other compounds listed in table 8A by selected ion monitoring GC/MS (GC/MS-SIM).

1.2 Table 1 lists the neutral, acidic, and basic organic compounds that can be determined by this method. The applicable concentration range of this method is compound and instrument dependent. Some compounds may require special treatment due to the limitations caused by sample preparation and/or chromatographic problems.

2.0 SUMMARY OF METHOD

2.1 This method is performed in accordance with the following extraction methodologies in SW846: 3510, 3520, 3546, 3550 and 3580.

2.2 The resultant methylene chloride extract is injected into a tuned and calibrated GC/MS system equipped with a fused silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

2.3 The peaks detected are identified qualitatively by comparison to characteristic ions and retention times specific to the known target list of compounds.

2.4 Once identified, the compound is quantitated by internal standard techniques with an average response factor generated from the calibration curve.

2.5 Additional unknown peaks with a response greater than 10 % of the closest internal standard may be processed through a library search with comparison to a NIST08 database. An estimated concentration is quantitated by assuming a response factor of 1.

2.6 This method includes analytical options for PAHs and other selected compounds by GC/MS-SIM. The extract is fortified with an additional SIM specific internal standard mix and analyzed using

selected ions that are characteristic of the compounds of interest following the analysis of lower concentration calibration standards analyzed under the same MS scan conditions. Qualitative and quantitative identification is conducted using the procedures employed for full scan analysis.

2.7 An option for preparing aqueous samples using a reduced initial volume (~250 ml) for analysis by a large volume injection (LVI) technique is also described.

3.0 REPORTING LIMIT & METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at or above the lowest concentration standard in the calibration curve, depending on the requirements of different regulatory programs. The RL must be above the established MDL. Detected concentrations below this concentration cannot be reported without qualification. See table 9 and 10. The RL concentrations are calculated for water matrix in ug/L using formula in sec. 13.4.1 and for soil matrix in ug/kg using formula in sec.13.4.2.

3.1.1 Compounds detected at concentrations between the reporting limit and MDL are quantitated and qualified as “J”, estimated value. Program or project specifications may dictate that “J” qualified compounds are not to be reported.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B, Revision 2.

3.2.1 Experimental MDLs must be determined in accordance with SGS SOP EQA075.

3.2.2 Process all raw data for the replicate analysis in each MDL study. Forward the processed data to the QA group for archiving.

4.0 DEFINITIONS

BATCH - a group of samples which behave similarly with respect to the sampling or the testing procedures being employed, and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

BLANK - an analytical sample designed to assess specific sources of laboratory contamination.

CONTINUING CALIBRATION - a mid-range calibration check standard run every 12 hours to verify the initial calibration of the system.

EXTRACTED ION CURRENT PROFILE (EICP) - a plot of ion abundance versus time (or scan number) for ion(s) of specified mass (Es).

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations which cover the working range of the instrument; used to define the linearity and dynamic range of the response of the mass spectrometer to the target compounds.

INTERNAL STANDARDS - compounds added to every standard, blank, matrix spike, matrix spike duplicate, and sample extract at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds and must be analytes that are not sample components.

MATRIX - the predominant material of which the sample to be analyzed is composed.

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards, is carried throughout the entire preparatory and analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD DETECTION LIMITS (MDLs) - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is distinguishable from method blank results.

PERCENT DIFFERENCE (%D) - As used to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

PRIMARY QUANTITATION ION - a contract specified ion used to quantitate a target analyte.

REAGENT WATER - water in which no interferant is observed at or above the minimum detection limit of the parameters of interest.

RECONSTRUCTED ION CHROMATOGRAM (RIC) - a mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

RELATIVE PERCENT DIFFERENCE (RPD) - As used to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

RESOLUTION - also termed separation or percent resolution, the separation between peaks on a chromatogram, calculated by dividing the depth of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

INITIAL CALIBRATUION VERIFICATION (SECOND SOURCE CALIBRATION STANDARD) - a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run whenever an initial calibration is performed.

SURROGATES - pure analytes added to every blank, sample, matrix spike, matrix spike duplicate, and standard in known amounts before extraction or other processing; used to evaluate analytical

efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Plan and Personal Protection Policy, which include the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets is made available to all personnel involved in these analyses.

5.3 The following analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, N-nitrosodimethylamine, and 4,4'-DDT. Prepare primary standards of these toxic compounds in a hood. A NIOSH/Mass approved toxic gas respirator must be worn when the analyst handles high concentrations of these toxic compounds.

6.0 INTERFERENCES

6.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

6.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other stages of sample processing. Refer to "The Preparation of Glassware for Extraction of organic contaminants" SOP for practices utilized in the extraction department.

6.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled.

6.4 To reduce carryover when high-concentration samples are sequentially analyzed, the syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it must be followed by the analysis of solvent to check for cross contamination.

7.0 SAMPLE COLLECTION, PRESERVATION, & HOLDING TIMES

7.1 Water samples may be collected in 1-liter or 250 ml-LVI glass bottles with Teflon insert in caps. Soil samples may be collected in 250-mL wide-mouth amber glass bottles.

7.1.1 Samples must be taken with care to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus avoiding possible phthalate contamination.

7.2 The samples must be protected from light and refrigerated at ≤6° C from the time of receipt until extraction and analysis.

7.3 Store the sample extracts at -10 °C in amber vials (protected from light), in sealed vials equipped with unpierced PTFE-lined septa.

7.5 HOLDING TIME

7.5.1 Aqueous samples must be extracted within 7 days of sampling.

7.5.2 Soil, sediments and concentrated waste samples must be extracted within 14 days of sampling.

7.5.3 Extracts must be analyzed within 40 days following extraction.

8.0 APPARATUS & MATERIALS

8.1 GAS CHROMATOGRAPH/MASS SPECTROMETER SYSTEM

8.1.1 Gas Chromatograph. HP-5890, HP-6890, Agilent 6890-N, or Agilent 7890 which includes an analytical system that is complete with a temperature programmable gas chromatograph and all required accessories including syringes, capillary chromatographic columns, and gases.

8.1.1.1 The injection port is designed for split or splitless injection with capillary columns.

8.1.1.2 The capillary column is directly coupled to the source.

8.1.2 Column.

8.1.2.1 30 m x 0.25 mm fused silica (0.25 μm film thickness) DB-5MS or equivalent capillary column. Condition the column as per manufacture's directions.

8.1.3 Mass Spectrometer (HP-5972, HP-5973 or Agilent 5975).

8.1.3.1 Full Scan Mode -Capable of acquiring mass spectra from mass/charge (m/z) 35-500 amu at a rate fast enough to acquire at least 5 (preferably 10 or more) mass spectra across each chromatographic peak of interest, using 70-volt (nominal) electron energy in the electron impact ionization mode.

8.1.3.2 SIM Mode- Capable of selective ion grouping at specified retention times for increased compound sensitivity (table 2a) or for applications requiring quantitation limits below the normal range.

8.1.3.3 Capable of producing a mass spectrum which meets all the EPA performance criteria in Table 3 when injecting 50 ng of Decafluorotriphenyl phosphine (DFTPP).

8.2 DATA SYSTEM

8.2.1 Acquisition and Instrument Control: HP Chemstation. A computer system is interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine readable media (disc) of all mass spectra obtained throughout the duration of the chromatographic program.

8.2.2 Data Processing: HP Enviroquant. The software accommodates searching of GC/MS data files for analytes which display specific fragmentation patterns. The software also allows integrating the abundance of an EICP between specified time or scan number limits. The data system includes the NIST08 spectra library for qualitative searches of non-target compounds present in the chromatogram. It flags all data files that have been edited manually by laboratory personnel.

8.2.3 Offline Magnetic Media Storage Device - the magnetic media storage device copies data for long term, offline storage.

8.3 SYRINGE

8.3.1 10 μL graduated, auto sampler (Hamilton or equiv.).

8.3.2 Micro liter syringes, various sizes

9.0 REAGENTS AND STANDARDS

9.1 Solvents - Ultra pure, chromatography grade methylene chloride and acetone.

9.2 Stock Standard Solutions.

9.2.1 Certified, commercially prepared standards, from two separate sources are used.

9.2.1.1 Base Neutrals.

- Base/Neutrals Mix #1 (Absolute: Semivolatile Organics Standard Mix # 1).
- Base/Neutrals Mix #2 (Absolute: Semivolatile Organics Standard Mix # 2).
- PAH Mix (Absolute: Semivolatile Organics Standard Mix # 7).
- PAH Mixture #2 (Ultra).
- PAH Selected Ion Monitoring Mixture
- Benzidines Mix (Absolute: Semivolatile Organics Standard Mix # 6).
- Toxic Substances #2 (Absolute: Semi volatile Organics Standard Mix # 5).
- Pyridines Mixture (Ultra).
- Additional requested compound(s) mix (Absolute).
- Base Neutral Mixture (2nd Source).

Acids

- Phenols Mix (Absolute: Semivolatile Organics Standard Mix # 8).
- Toxic Substances #1(Absolute: Semivolatile Organics Standard Mix # 4).
- Acid Mixture (2nd Source). Internal Standard Mixtures.

9.2.2 Internal Standard Mixtures

9.2.2.1 Ultra (or equivalent) at a concentration of 4,000 μg/mL for each of the following compounds:

Full Scan

- 1,4-Dichlorobenzene-d4
- Naphthalene-d8
- Acenaphthene-d10
- Phenanthrene-d10
- Chrysene-d12
- Perylene-d12

SIM

- 1,2-Dichlorobenzene-d4
- 1-Methylnaphthalene-d10
- Fluorene-d10
- Fluoranthene-d10
- Benzo(a)pyrene-d12
- 1,4-Dioxane-d8 (for Isotopic Dilution analysis of 1,4 Dioxane)

9.2.2.2 The internal standards must permit most of the components of interest in a chromatogram to have retention times of 0.8 - 1.20 relative to one of the internal standards.

9.2.2.3 Each 1 mL sample extract, and standard undergoing analysis must be spiked with 10 µl of the internal standard mixtures, resulting in a concentration of 40 µg/mL of each internal standard for full scan analysis and 4µg/mL for SIM analysis. 20 ug/mL of 1,4-Dioxane-d8 is added during extraction for Isotopic Dilution analysis of 1,4-Dioxane via SIM. For LVI, each sample extract will be spiked with 2 µl of the internal standard mixtures.

9.2.3 Surrogate Standard Mixture.

9.2.3.1 B/N Surrogate Standard Mix: RESTEK (or equivalent) at a concentration of 5,000 µg/mL each surrogate compound.

- Nitrobenzene-d5.
- 2-Fluorobiphenyl.
- p-Terphenyl-d14.

9.2.3.1.1 For DOD PAH target analytes by SIM; additional surrogates are required.

- Fluoranthene-d_{10}
- 2-methylnaphthalene-d_{10}

9.2.3.2 Acid Surrogate Standard Mix: RESTEK (or equivalent) at a concentration of 7,500 µg/mL each surrogate compound.

- Phenol-d5.
- 2-Fluorophenol.
- 2,4,6-Tribromophenol.

9.2.4 DFTPP Tune Stock.

9.2.4.1 Protocol (or equivalent) at a concentration of 2,500 μg/mL for the following compounds.

- Decafluorotriphenylphosphine.
- 4,4'-DDT.
- Benzidine.
- Pentachlorophenol.

9.2.5 Store standards at -10°C or less when not in use or according to the manufacturer's documented holding time and storage temperature recommendations. Stock standard solutions must be replaced after 1 year or sooner if manufacture's expiration date comes first or comparison with quality control check samples indicates degradation.

9.3 Surrogate Spiking Solutions.

9.3.1 One surrogate spiking solution, containing both base/neutral and acid surrogates at a concentration of 50μg/mL is prepared in extractions. Spike each sample, and blank with 1 mL of solution prior to extraction, for a final concentration of 50 μg/l of each surrogate compound in the extract. For LVI spike, each sample with 200 μl of surrogate solution.

9.3.2 A calibration range must be constructed for the surrogate compounds. Accordingly, appropriate amounts of surrogates are mixed with each calibration solution to define a range similar to the target compounds.

9.3.3 Store at -10 °C or less or according to the manufacturer's documented storage temperature recommendations. Prepare fresh surrogate spiking solutions every six months, or sooner, if the manufacturer's expiration dates come first or if the solution has degraded or evaporated.

9.4 Intermediate Calibration Standard Solution.

9.4.1 The calibration stock solution is prepared by adding an appropriate amount of each stock and surrogate compounds into a 10 mL volumetric flask. Dilute the solution to the volume with methylene chloride and mix thoroughly. Refer to Table 7A for details.

9.5 Calibration Standards.

9.5.1 Initial Calibration Standards.

9.5.1.1 Calibration standards containing the surrogate compounds must be made by quantitative dilutions of the above intermediate solution. The calibration standards are prepared at a minimum of five concentrations to cover the range of 1 - 100 μg/mL for full scan, 0.2 – 20 μg/mL for LVI, and 0.02 – 5ug/mL for SIM, 0.002 – 2 ug/mL for SIM LVI,0.02—20 ug/ml for 1,4-Dioxane by SIM isotopic dilution, depending upon project specific requirements. Suggested levels and preparations are shown in Table 7A and 7B.

9.5.2 Continuing Calibration Verification.

9.5.2.1 The concentration of the mid range standard used for continuing calibration verification is 50 μg/mL for full scan, 10 μg/mL for LVI, 1.0 μg/mL for SIM, and 0.2 μg/mL for SIM LVI.

9.5.3 Store the calibration standards in a refrigerator at ≤ 6 °C and prepare every 6 months or before the manufacturer's expiration date, whichever is sooner. Standards must be replaced immediately if the analysis of check standards indicates degradation.

9.6 Initial Calibration verification (ICV) -Second source calibration check standard.

9.6.1 The ICV standard is prepared per Table 7E, using the intermediate solutions prepared in Extraction. The ICV must be prepared from a second manufacturer or from a manufacturer's lot prepared independently from the lot used for calibration.

9.6.2 The ICV is analyzed after each initial calibration.

9.7 GC/MS Performance Checks.

9.7.1 The solution is prepared at 50 μg/mL by making a 1:50 dilution of DFTPP stock solution (Section 9.2.4) in methylene chloride.

9.8 Matrix Spike Solutions.

9.8.1 The matrix spike solutions for both Base/Neutral and Acid are prepared in Acetone at a concentration of 100 μg/mL for each compound. Prepare the matrix spike, matrix spike duplicate and blank spike by spiking the selected sample and the blank with 1 mL of these solutions for a final concentration of 50 μg/l of each compound. MS/MSD and BS must be prepared with target analytes from the same source as used for the initial calibration. For LVI, spike with 200 μl of the spiking solution

9.9 All organic new standard solutions are analyzed prior to use to verify the accuracy of the prepared concentration.

9.9.1 The prepared standard solution is analyzed using the determinative (instrumental) technique for the method.

9.9.2 The solution is analyzed following the completion of instrument calibration or a calibration check.

9.9.3 The concentration of the standard solution is determined using the software routines used in determining the acceptability of calibration verification.

9.9.4 The data is evaluated, and the percent difference determined. The standard solution is approved for use if all designated compounds are present in the solution and the percent difference is less than the established criteria (±20%).

10.0 CALIBRATION

10.1 Initial Calibration.

10.1.1 The calibration range covered for routine analysis under RCRA employs standards of 1, 2, 5, 10, 25, 50, 80, 100 µg/mL for full scan, 0.2, 0.4, 1, 2, 5, 10, 16, 20 µg/mL for LVI, and 0.01 (bis(2-chloroethyl) ether (BCEE) only) 0.02, 0.05, 0.10, 0.20, 0.5, 1.0, 2.5, 5.0 µg/mL for SIM, and 0.002, 0.004, 0.01, 0.02, 0.04, 0.1, 0.2, 0.5, 1.0, 2.0 µg/mL for SIM LVI; 0.02, 0.01, 0.05, 0.1, 0.2, 0.5, 1.0, 5.0, 10.0, 20.0 ug/ml for 1,4-dioxane by SIM isotopic dilution. A minimum of five standards must be run sequentially. The reporting limit is established by the concentration of the lowest standard analyzed during the initial calibration. Lower concentration standard may be needed to meet the reporting limit requirements of state specific regulatory program. The linear range covered by this calibration is the highest concentration standard.

10.1.2 A calibration range must be constructed for each surrogate compound. Accordingly, add appropriate amounts of surrogate spiking solutions to the calibration solution to define a range similar to the target compounds.

10.1.3 Aliquot 1 mL of each calibration standard into a 2 mL crimp top vial.

10.1.4 Prior to analysis, add 10 µL of the applicable (Full scan and/or SIM) internal standard solution (Section 9.2.2) to each standard. This results in a concentration of 40 µg/mL (Full scan) and 4ug/mL (SIM) for each internal standard. For 1,4-Dioxane isotope dilution analysis, add 1mL of 20µg/mL 1,4-Dioxane-d8 prior to extraction of samples.

10.1.5 Analyze the standard solutions using the conditions established in Section 11.0. Each analyte is quantitatively determined by internal standard technique using the closest eluting internal standard and the corresponding area of the major ion. See Table 6. 1,4-Dioxane via SIM is analyzed using Isotope Dilution.

10.1.6 The Response Factor (RF) is defined in Section 13.1. Calculate the mean RF for each target analyte, using minimum of five RF values calculated from the initial calibration curve.

10.1.7 For the initial calibration to be valid, the following criteria must be met.

10.1.7.1 The percent relative standard deviation (% RSD) (see Section 13.2) of all target analytes must be less than or equal to 20%.

10.1.7.1.1 If the average response factor criteria cannot be achieved, employ an alternative calibration linearity model. Do not force the regression line through the origin and do not employ 0,0 as a sixth calibration standard. The correlation coefficient (r value) must be $\geq$ 0.995 or ±0.99 (for r^2 value) for each compound to be acceptable, and calibration curves using quadratic model must have minimum 6 calibration points.

10.1.7.1.2 In some cases, although the %RSD of a target analyte is≤ 20%, more suitable calibration model may be employed or “best fit” rather than average calibration or response factor. Choice of calibration model can be based on previous experience with the same analyte or knowledge of the physics of the detector on the instrument where the calibration is performed. To determine the suitability of linear or quadratic calibration, the %RE is calculated, where the calibration data is refitted back to the curve (see section 10.1.7.1.3 for calculation and criteria). The choice of

calibration model must not be used to compensate for detector saturation or to avoid proper instrument maintenance.

10.1.7.1.3 The calculation of relative error (%RE) must be performed for two calibration levels, the standard at or near the mid-point of the initial calibration and the standard at the lowest level.

Relative Error is calculated using the following equation:

$$\% \text{ Relative Error} = \frac{x'i - xi}{Xi} \times 100$$

xi = True value for the calibration standard
x'i = Measured concentration of the calibration standard

The Relative Error at lowest level must be within ±50% from the true value. The RE for mid-point level must be within ±30% from the true value. Both levels after calculating %RE must be fitted back into the curve.

10.1.7.2.4 If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient for the linear calibration option, then the chromatographic system is considered too reactive for the analysis to begin. Perform corrective action and recalibrate if the calibration criteria cannot be achieved.

10.1.7.2 Below is a table containing minimum RFs that may be used as guidance in determining if the system is behaving properly and as a check to see if calibration standards are prepared correctly. The minimum RFs are determined using not only specific ions but also specific instrument conditions, which may vary from one instrument to another. It is recommended to compare the minimum RFs for target analytes from previous initial calibrations to the minimum RFs form current initial calibration on each instrument to make sure the RFs are stable.
Note: For target analyte, whose RF<0.01 (response of peak is <1/100 the response of the Internal Standard), it is recommended that its concentration in relation to other analytes be increased to make the response more comparable to other analytes

Guidance Response Factor Table for both scan and SIM mode analysis

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis (2-chloroethyl) ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500

Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-hloroethoxy) methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrobenzene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010

Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl) phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd) pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
1,4 Dioxane	0.010
2,3,4,6-Tetrachlorophenol	0.010

10.1.7.3.1 Due to the large number of compounds, some compounds will fail to meet the minimum response factor criteria. They may be used as qualified data or estimated values for screening purposes. Non-detects may be reported if adequate sensitivity has been demonstrated at the applicable lower quantitation limit.

10.1.7.4 The initial calibration criteria for this method apply to all additional compounds of concern specified by the client.

10.1.7.5 Structural isomers that produce very similar mass spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is more than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution must be verified on the mid - point concentration of the initial calibration (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene). Print the check and keep it on file.

10.1.8 The laboratory may remove individual analyte calibration levels from the lowest and/or highest levels of the curve. Multiple levels may be removed, but removal of interior levels is not permitted.

10.1.8.1 The laboratory may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. If a calibration standard was removed from the interior of the calibration, this particular standard calibration level must be removed for all analytes. Removal of calibration points from the interior of the curve is not to be used to compensate for lack of maintenance or repair to the instrument.

10.1.8.1.1 The laboratory must adjust the LOQ/reporting limit and quantitation range of the calibration based on the concentration of the remaining high and low calibration standards.

10.1.8.1.2 The laboratory must ensure that the remaining initial calibration standards are sufficient to meet the minimum requirements for

number of initial calibration points as mandated by the method, or regulatory requirements.

10.1.8.1.3 The laboratory may replace a calibration standard provided that:

a) the laboratory analyzes the replacement standard within twenty-four (24) hours of the original calibration standard analysis for that particular calibration level;

b) the laboratory replaces all analytes of the replacement calibration standard if a standard within the interior of the calibration is replaced; and

c) the laboratory limits the replacement of calibration standards to one calibration standard concentration.

10.1.8.1.4 The laboratory must document a technically valid reason for either removal or replacement of any interior calibration point.

10.2 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard.

10.2.1 The calibration is verified with a calibration check standard at 50 μg/mL (Full scan) or 1 ug/mL (SIM) from a secondary source (Section 9.6). It must be analyzed immediately following the initial calibration.

10.2.2 The percent difference (% D) (Section 13.3) for this standard must meet the criteria of 30% for all the target compounds.

10.2.2.1 If % D is greater than 30%, reanalyze the second source check. If the criteria cannot be met upon re-injection, re-prepare the second source solution using a fresh ampoule and repeat the process.

10.2.2.2 If the %D criteria cannot be achieved after re-preparation of the second source, prepare a third source and repeat the process. Make fresh calibration standards using one of the two standard sources that match each other.

10.3 Continuing Calibration Verification Standard - CCV

10.3.1 A calibration verification standard at close mid-level concentration of the initial calibration range at 50 ug/mL for full scan, 10 ug/mL for full scan LVI, 1 ug/mL for SIM, and 0.2 ug/mL for LVI SIM must be acquired every 12 hrs. The 12-hour clock starts at the time of injection of daily CCV.

10.3.1.1 The calibration verification standard selected must be near concentration of the midpoint calibration standard or near the action level for the project specified.

10.3.2 For the continuing calibration to be valid, all of the following specified criteria must be met.

10.3.2.1 Each of the most common target analytes in the calibration verification standard must meet the minimum response factors as noted in the Minimum Response Factor Table in section 10.1.7.3.

10.3.2.2 All target compounds of interest must be evaluated using 20% D criteria. If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid.

10.3.2.3 Due to the large numbers of compounds that may be analyzed by this method, it is expected that some compounds will fail to meet the 20% D criterion. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples.

10.3.2.4 In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to the compound at the applicable quantitation limits. For situation when the failed compound is present, the concentration must be reported as estimated.

10.3.2.4.1 Compounds with response factors that exceed the 20% D in the CCV compared to the initial calibration with high bias may be still reported when as non-detect, or in cases where the failed compound is present, the concentration must be reported as estimated value.

10.3.2.4.2 Compounds that do not meet the 20% D in the CCV compared to the initial calibration due to low response factors can only be reported if the low sensitivity of the instrument is still achieved. This sensitivity must be verified by running a low-level standard check at reporting limit. If a positive result for the compound is found, then adequate sensitivity has been demonstrated and the run can proceed.

10.3.2.5 The resolution check for structural isomers must be verified for each CCV standard Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise structural isomers are identified as isomeric pairs. Print the check and keep it on file.

10.3.3 If the first continuing calibration verification does not meet criteria, a second standard may be injected after notifying the team leader/manager and checking the system for defects.

10.3.3.1 A continuing calibration check is allowed to be repeated only once; if the second trial fails, a new initial calibration must be performed or refer to section 10.3.2.4. In situations where the first check fails to meet the criteria, the instrument logbook must have clearly documented notations as to what the problem was and what corrective action was implemented to enable the second check to pass.

10.3.4 If the verification criteria cannot be achieved, a new initial calibration must be performed or refer to section 10.3.2.4.

10.3.5 If any of the internal standard areas change by a factor of two (- 50% to + 100%) or the retention time changes by more than 30 seconds from the midpoint standard of the last initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate.

10.3.5.1 Reanalyze the continuing calibration standard. New initial calibration is required if reanalyzed standard continues to fail the internal standard requirements.

10.3.5.2 All samples analyzed while the system was out of control must be reanalyzed following corrective action.

11.0 PROCEDURE

11.1 Instrument Conditions.

11.1.1 Recommended instrument conditions are listed in Table 2 and 2a (SIM only). Modifications of parameters specified with an asterisk are allowed as long as criteria of calibration are met. Any modification must be approved by team leader/manger. DFTPP, Standards, QC and samples must all be run under the exact same operating conditions, including EM voltage.

11.2 GC/MS Performance Checks.

11.2.1 Mass Spectrometer Tuning. Inject 1 μL of 50 ng/μL DFTPP solution directly on to the column.

11.2.2 The GC/MS system must be checked to verify that acceptable performance criteria are achieved (see Table 3).

11.2.3 **This performance test must be passed before every initial calibration curve.** Evaluate the tune spectrum using three mass scans from the chromatographic peak and a subtraction of instrument background.

11.2.3.1 Select the scans at the peak apex and one to each side of the apex.

11.2.3.2 Calculate an average of the mass abundances from the three scans.

11.2.3.3 Background subtraction is required. Select a single scan in the chromatogram that is absent of any interfering compound peak and acquired within no more than 20 scans to the elution of DFTPP. The background subtraction must be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tuning compound peak.

11.2.4 If all the criteria are not achieved, the analyst must retune the mass spectrometer with team leader/manager and repeat the test until all criteria are met.

11.2.4.1 Alternatively, an additional scan on each side of the peak apex may be selected and included in the averaging of the mass scans. This will provide a mass spectrum of five averaged scans centered on the peak apex. NOTE: The selection of additional mass scans for tuning may only be performed with supervisory approval on a case by case basis.

11.2.5 In order to assess GC column performance and injection port inertness, the DFTPP tune standard also contains appropriate amount of 4,4'-DDT, benzidine and pentachlorophenol.

11.2.6 All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

11.2.6.1 Injection Port Inertness Check.

11.2.6.1.1 The injection port inertness of the GC portion of the GC/MS is evaluated by the percent breakdown of 4,4'-DDT. DDT is easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated by high boiling residue from sample injection or when the injector contains metal fittings. Check for degradation problems by injecting a GC/MS tune standard containing 4,4'-DDT , regardless of whether DDT is a target analyte. The degradation of DDT to DDE and DDD must not exceed 20%, in order to proceed with calibration procedures. Refer to Section 13.7 for calculation. Print the check and keep it on file.

11.2.6.2 Column Performance Check.

11.2.6.2.1 The condition of the GC column is evaluated by the tailing of benzidine and pentachlorophenol. Benzidine and pentachlorophenol must be present at their normal responses, with no visible peak tailing, as demonstrated by the peak tailing factors. The tailing factor criteria for benzidine (base-neutral fraction) must be $\leq$ 2 and for pentachlorophenol (acid fraction) must be $\leq$ 2. Print the check daily and keep on file:

11.2.6.3 If degradation is excessive and/or poor chromatography is observed, the injector port may require cleaning. It may also be necessary to break off the first 6-12 in. of the capillary column.

11.3 Initial Calibration

11.3.1 Refer to Section 10.1.

11.4 Initial calibration Verification (ICV) - Second Source Calibration Check

11.4.1 This standard must at least be analyzed when initial calibration provided. Refer to Section 10.2.

11.5 Continuing Calibration Checks

11.5.1 Refer to Section 10.3.

11.6 Sample Analysis.

11.6.1 Allow the sample extract (preparation procedure detailed in SOP EOP001, 003 or 3546) to warm to room temperature. Spike 10 μl of the appropriate internal standard mix (4,000 μg/mL for full scan and 400ug/mL for SIM) into 1 mL of sample extract, just prior to analysis. This is equivalent to a concentration of 40 μg/mL (full scan) and 4ug/mL (SIM) of each internal standard.1,4-Dioxane-d8 is added prior to extraction for isotopic dilution. For LVI, each sample extract will be spiked with 2 μl of internal standard mixtures.

11.6.2 Inject 1 μL aliquot of the sample extract into the GC/MS system. A split injection technology is used.

11.6.3 If the responses for any of the ions of interest exceed the working range of the GC/MS system, dilute a stored extract if available and reanalyze.

11.6.4 When the extracts are not being used for the analysis, store them at -10°C, protected from light, in sealed vials equipped with unpierced PTFE-lined septa.

11.7 Sample Dilution

11.7.1 Establish dilutions of samples in order for detected targets to fall within the calibration range or to minimize matrix interference.

- Utilize screen data (specific project only).
- Utilize acquired sample data.
- Utilize the history program or approval from client/project.
- Sample characteristics (appearance, odor).

11.7.2 If no lower dilution has been reported, the dilution factor chosen must keep the response of the largest peak for a target analyte in the upper half of the initial calibration range of the instrument.

11.7.3 Preparing Dilutions.

11.7.3.1 Prepare sample dilutions quantitatively. Dilute the sample extract with methylene chloride using logical volume to volume ratios, i.e., 1:5, 1:10, 1:50, etc. Large dilutions may require serial dilutions or the use of a Class A 10 mL volumetric flask.

11.7.3.2 Syringe dilutions. – Calibrated syringes are used to prepare dilutions. Add the appropriate amount of methylene chloride to a clean autosampler vial. Add the proper amount of sample using a calibrated syringe of the appropriate volume for the dilution. Add sufficient internal standard to maintain a concentration of 40ug/mL. Cap the vial and gently shake to disperse the sample through the solvent.

11.7.3.3 Volumetric Flask Dilutions – Large dilutions may require the use of a 10 mL or larger Class A Volumetric flask.

11.8 Establishing Search Criteria for target compounds. Search criteria for each compound listed in the method must be entered into the method quantitation/identification file in the Enviroquant software package. This activity must be performed before attempting qualitative and

quantitative analysis on any acquired data file. The search criteria are based on compound retention time and the characteristic ions from the reference mass spectrum. Characteristic ions are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. The number of secondary ions displayed for each compound search varies between compounds.

11.8.1 Select the primary ion for the target compound from the characteristic ions in Table 6. If multiple characteristic ions are listed, the first ion is the major (primary) ion. Enter this ion as the search ion. Enter the relative abundance of this ion (100% for base peak ions) and set the relative abundance window at ± 30%.

11.8.1.2 Alternate primary ions may be selected when interferences exist from ion abundance contribution from close eluting compounds.

11.8.2 Enter the remaining ions as secondary ions. Secondary ions are not be used to locate peaks within the search window but are be used to support the qualitative identification of selected peaks. The number of secondary ions displayed for each compound search varies between compounds depending on the number of ions in the spectra >30% relative abundance.

11.8.3 Set the relative abundance windows for the secondary ions at ± 30%.

11.8.4 The qualitative identification of compounds determined by this method is based on retention time (RT). The RT must be within ±10 seconds of the RT for this analyte in the CCV run at the beginning of the 12-hour period (delta RT0.17 minute) or within ±10 seconds relative to the shift of the associated internal standard (delta RT of the IS±10 seconds).

11.9 Data Interpretation.

11.9.1 Executing Qualitative Searches. The target compounds shall be identified by analyst with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound.

11.9.1.1 The search procedure will identify peaks within the search window using the primary ion only. Secondary ions and the relative retention are used to determine “the best match”. If the best match contains secondary ions outside the relative abundance window, they will be flagged with a # sign.

11.9.2 Qualitative Identification. The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. Compounds are identified when the following criteria are met.

11.9.2.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other.

11.9.2.2 The absolute retention time (RT)must be within ±10 seconds of the retention time for this analyte in the daily standard (CCV) run at the beginning of the 12-hour period (delta RT 0.17 minute) or within ±10 seconds relative to the shift of the

associate internal standard (IS) (delta RT of the IS ± 10 seconds). If RT drift is significant, relative retention time (RRT) may be used as an alternative to delta RTs. The RRT of the analyte in the sample should be within RRT limits of the analyte in the standards.

$$RRT = \frac{\text{RT of the analyte}}{\text{RT of the IS}}$$

11.9.2.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

11.9.2.3.1 If a chromatographic peak exhibits a spectrum containing an ion with relative abundance outside the relative abundance window is selected for reporting, the analyst must annotate the spectra that the compound qualified based on his/her best judgement. This circumstance will most often occur from coeluting compounds with similar ions or background matrix interferences.

11.9.3 Quantitative Analysis.

11.9.3.1 Once a target compound has been identified, its concentration (Section 13.4) will be based on the integrated area of the quantitation ion, normally the base peak (Table 6). The compound is quantitated by internal standard technique with an average response factor generated from the initial calibration curve. 1,4-Dioxane via SIM is analyzed via Isotope Dilution.

11.9.3.2 If the sample produces interference for the primary ion, use a secondary ion to quantitate. This may be characterized by an excessive background signal of the same ion, which distorts the peak shape beyond a definitive integration. Also, interference could severely inhibit the response of the internal standard ion. The secondary ion must be used to generate a new response factor.

11.10 Library Search for Tentatively Identified Compounds.

11.10.1 If a library search is requested, the analyst must perform a forward library search of the NIST08 mass spectral library to tentatively identify 10 to 15 non-reported compounds (15 for base, 10 for acid, 25 for base/acid fraction).

11.10.2 Guidelines for making tentative identification are listed below.

11.10.2.1 These compounds must have a response greater than 10% of the nearest internal standard. The response is obtained from the integration for peak area of the Total Ion Chromatogram (TIC).

11.10.2.2 The search is to include a spectral printout of the 3 best library matches for a particular substance. The results are to be interpreted by analyst.

11.10.2.3 Molecular ions present in the reference spectrum must be present in the sample spectrum.

11.10.2.4 Relative intensities of major ions in the reference spectrum (ions > 10 % of the most abundant ion) must be present in the sample spectrum.

11.10.2.5 The relative intensities of the major ions must agree within ± 20 %. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must between 30 and 70%).

11.10.2.6 Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.

11.10.2.7 Ions present in the reference spectrum but not in the sample spectrum must be verified by performing further manual background subtraction to eliminate the interference created by coeluting peaks and/or matrix interference.

11.10.3 Quantitation of the tentatively identified compounds is obtained from the total ion chromatogram based on a response factor of 1 and is to be tabulated on the library search summary data sheet.

11.10.4 The resulting concentration must be reported indicating: (1) that the value is estimate, and (2) which internal standard was used to determine concentration. Quantitation is performed on the nearest internal standard.

11.10.5 Peaks that are suspected to be aldol-condensation reaction products (i.e., 4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) shall be searched and reported but not counted towards the total TIC count.

11.10.6 Any peak naming as “System artifact” (from the column bleedings) or “Internal Standard” (added by lab for other test, like SIM analysis) shall be searched and reported but not counted towards the total TIC count.

11.11 Selected Ion Monitoring (SIM) Option

NOTE: The use of SIM is not allowed by the SCDHEC for samples from South Carolina.

11.11.1 Instrument Set-Up: Modify the method for SIM analysis and define ion groups with retention times, ions and dwell times to include base peak ion for the target compounds of interest, surrogates, and internal standards (Table 2a, Table 8a). Select a mass dwell time of 50 milliseconds for all compounds.

11.11.2 Calibration: Calibrate the mass spectrometer in the selected ion monitoring mode using 7 calibration standards of 0.02, 0.05, 0.10, 0.20, 1.0, 2.5, 5.0 µg/ml. Use 0.02, 0.05, 0.1, 0.2, 0.5, 1.0, 2.5, 5.0, 10.0, 20.0 ug/ml for calibration of 1,4-Dioxane by SIM isotopic dilution. Spike each standard with the SIM specific internal standard solution at 4ug/mL (1,4-Dioxane-d8 is calibrated using 20ug/mL). Calculate individual response factors and response factor RSDs using the procedures and criteria described in Section 10.1.6, 10.1.7.3 and 10.1.7.4.

11.11.3 Initial Calibration Verification. Verify the initial calibration after its completion using a 1.0 µg/mL calibration standard purchased or prepared from a second standards reference materials source. The initial calibration verification must meet the criteria of Section 10.2.2.

11.11.4 Continuing Calibration Verification. Verify the initial calibration every 12 hours using a 1.0 or 0.5 µg/mL calibration. The continuing calibration verification must meet the criteria of Section 10.3.

11.11.5 Sample Extract Analysis: Each extract has been previously spiked with the SIM internal standard at 4µg/mL. Analyze the sample extracts for the compounds of interest using the SIM scan parameters employed for the calibration standards.

11.11.6 Surrogate Standard Calculation. Report surrogate spike accuracy for the surrogates spiked for the full scan GC/MS analysis at 50µg/ml.

12.0 QUALITY CONTROL

12.1 QC Requirements Summary.

Daily GC/MS Performance Checks	Before initial calibration
Initial Calibration	Whenever needed.
Second Source Calibration Check	Following initial calibration
Continuing Calibration Verification	Every 12 hours.
Method Blank	One per extraction batch*.
Blank Spike	One per extraction batch*.
Matrix Spike	One per extraction batch*.
Matrix Spike Duplicate	One per extraction batch*.
Surrogate	Every sample extract and standard.
Internal Standard	Every sample extract and standard.

*The maximum number of samples per batch is twenty or per project specification.

12.2 Daily GC/MS Performance Checks.

12.2.1 Refer to Section 11.2.

12.3 Initial Calibration.

12.3.1 Refer to Section 10.1.

12.4 Initial Calibration Verification (ICV) - Second Source Calibration Check.

12.4.1 Refer to Section 10.2.

12.5 Continuing Calibration Verification.

12.5.1 Refer to section 10.3.

12.6 Method blank.

12.6.1 The method blank is either reagent water or anhydrous sodium sulfate (depending on the sample matrix) which must be extracted with each set of 20 or less samples. For a running batch, a new method blank is required for each different extraction day. The method blank is then extracted and carried through all stages of the sample preparation and measurement.

12.6.2 If the method blank contains a target analyte above its MDL, the entire batch must be re-extracted and re-analyzed.

12.6.2.1 A method blank may be acceptable if it is less than 5% (20x) of the sample result for the same analyte.

12.6.3 Surrogate compounds are added to the method blank prior to extraction. If the surrogate accuracy in the method blank does not meet in house criteria, it must be reanalyzed. If the reanalysis confirms the original data, the entire batch must be re-extracted.

12.7 Blank Spike

12.7.1 A blank spike must be extracted with each set of 20 or less samples. For a running batch, a new blank spike is required for each different extraction day. The blank spike consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same volume. It is spiked with the same analytes at the same concentrations as the matrix spike/matrix spike duplicate.

12.7.2 The blank spike recoveries must be assessed using laboratory in house limits.

12.7.3 If a blank spike is out of control, the following corrective actions must be taken, and all the associated samples must be re-extracted and reanalyzed. The exception is if the blank spike recovery is high and no hits reported in associated samples and QC batch. In that case, the sample results can be reported with footnote (remark) and no further action is required.

12.7.3.1 Check to be sure that there are no errors in the calculations, or spike solutions. If errors are found, recalculate the data accordingly.

12.7.3.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample batch.

12.7.3.3 If no problem is found, re-extract and reanalyze the sample batch.

12.8 Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

12.8.1 One sample is randomly selected from each extraction batch and spiked in duplicate to assess the performance of the method as applied to a particular matrix and to provide information on the homogeneity of the matrix. Both the MS and MSD are carried through the complete sample preparation, and determinative procedures.

12.8.2 Matrix spikes are prepared by spiking an actual sample at a concentration of 50 μg/l for both base/neutral and acids.

12.8.3 Assess the matrix spike recoveries (% R) (Section 13.5) and relative percent difference (RPD) (Section 13.6) against the in-house control limits.

12.8.4 If the matrix spike accuracy of any individual compound is out of control, the accuracy for the compound in the blank spike must be within control. In such case, matrix interference is assumed, and the data is reported with footnote (e.g., spike recovery indicates possible matrix interference). No further corrective action is required.

12.9 Surrogates

12.9.1 All standards, blanks, sample extracts, and matrix spikes contain surrogate compounds which are used to monitor the performance of the extraction and analytical system.

12.9.2 The recoveries (Section 13.5) of the surrogates must be evaluated to determine whether or not they fall within surrogate control limits developed by the laboratory annually.

12.9.3 If the recovery of any surrogate compound does not meet the control limits, the calculation must be checked for possible error. The surrogate solution must be checked for degradation. Contamination and instrument performance must also be reviewed.

12.9.3.1 Reanalyze the extract if no calculation errors are detected. If the surrogate recoveries for the reanalyzed extract are in control, report the data from the reanalysis only.

12.9.3.2 If the data from the reanalysis is also out of control, re-extract and reanalyze the sample. The testing of concrete or gravel samples may result in low acid fraction surrogate recovery and re-extraction is not necessary. Surrogates will be properly footnoted in the report to reflect this.

12.9.3.3 If, upon reanalysis, the surrogate recoveries are acceptable, report the reanalysis data. If the holding time has expired prior to the reanalysis, report both the original and reanalysis results and note the holding time problem.

12.9.3.4 If the recovery is again not within limits, the problem is considered to be matrix interference. Submit both data sets with the original analysis being reported.

12.9.4 If the sample exhibits matrix interference, defined as excessive signal where target or non-target responses are greater than the response of the internal standards. In this case, reanalysis may not be required following team leader/manager approval; the surrogates will be qualified as outside the limits due to matrix interference. Alternatively, sample may be reanalyzed on dilution, if the reanalysis is again not within the limit, the sample must be reported with a footnote indicating that there was possible matrix interference.

12.9.5 If any two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective active is necessary.

12.10 Internal Standards.

12.10.1 Retention time for all internal standards must be within ± 30 seconds of the corresponding internal standard in the latest continuing calibration or 50 μg/mL standard of initial calibration.

12.10.2 The area (Extracted Ion Current Profile) of the internal standard in all analyses must be within 50 to 200 % of the corresponding area of the latest calibration standard (12 hr. time period).

12.10.3 If the area of internal standard does not meet control limits, the calculations must be checked. If a problem is not discovered, the sample must be reanalyzed.

12.10.4 If the areas are acceptable upon reanalysis, the reanalysis data is reported.

12.10.5 If the areas are unacceptable upon reanalysis, then both sets of data are submitted with the original analysis reported.

12.11 Refer to Project Specific Bench Notes (MS8270) for additional program or client specific QC Requirements

13.0 CALCULATION

13.1 Response Factor (RF).

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:
A_s = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_s = Concentration of the compound being measured (μg/mL).
C_{is} = Concentration of the specific internal standard (μg/mL).

13.2 Percent Relative Standard Deviation (%RSD).

$$\%RSD = \frac{SD}{RF_{av}} \times 100$$

where:
SD = Standard Deviation.
RF_{av} = Average response factor from initial calibration.

13.3 Percent Difference (%D).

$$\% D = \frac{| RF_{av} - RF_{cv} |}{RF_{av}} X\ 100$$

where: RF_{cv} = Response factor from Calibration Verification Standard.

13.4 Concentration (Conc.).

13.4.1 for water:

$$\text{Conc. } (\mu g/l) = \frac{A_s \times C_{is} \times V_f \times D \times 1000}{A_{is} \times RF_{av} \times V_i}$$

13.4.2 for soil/sediment (on a dry weight basis):

$$\text{Conc. } (\mu g/kg) = \frac{A_s \times C_{is} \times V_f \times D \times 1000}{A_{is} \times RF_{av} \times W_s \times S}$$

where:
V_f = Final Volume of total extract (mL).
D = Secondary dilution factor.
V_i = Initial volume of water extracted (mL).
W_s = Weight of sample extracted (g).
S = (100 - % moisture in sample) / 100.

13.5 Percent Recovery (%R).

$$\%\ R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

13.6 Relative Percent Difference (RPD).

$$RPD = \frac{|MSC - SDC|}{(1/2)(MSC+MSDC)} \times 100$$

where:

MSC = Matrix Spike Concentration.
MSDC = Matrix Spike Duplicate Concentration.

13.7 Percent Breakdown.

$$\%\ \text{Breakdown for DDT} = \frac{\text{Total DDT degradation peak area}}{\text{Total DDT peak area}} \times 100$$

where:
Total DDT degradation peak area = DDE + DDD
Total DDT peak area = DDT + DDE + DDD.

13.8 Linear regression by the internal standard technique.

$$C_s = \left(\frac{\frac{A_s}{A_{is}} - b}{a} \right) \times C_{is}$$

Where:
Cs = concentration of target analyte
As = Area of target analyte
Cis = concentration of the internal standard
b = Intercept
a = slope of the line

$$a = \frac{N \Sigma xy - \Sigma x \Sigma y}{N \Sigma x^2 - (\Sigma x)^2}$$

$$b = \frac{\Sigma y - a \Sigma x}{N}$$

N = number of points
x = amount of analyte
y = response of instrument

13.9 Quadratic curve with internal standard technique

$$Cs = \frac{-b \pm \sqrt{b^2 - 4a\left(c - \frac{A_s \times C_{is}}{A_{is}}\right)}}{2a}$$

Where:
Cs = concentration of target analyte
As = Area of target analyte
Cis = concentration of the internal standard
b = Intercept
a = slope of the line

13.10 Correlation Coefficient

$$r = \frac{\Sigma(x - \bar{x})(y - \bar{y})}{\sqrt{\Sigma(x - \bar{x})^2 \Sigma(y - \bar{y})^2}}$$

Where r = correlation coefficient
x = amount of analyte
y = response of instrument

$\bar{x}$ = average of x values

$\bar{y}$ = average of y values

14.0 DOCUMENTATION

14.1 The Analytical Logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

14.1.1 If samples require reanalysis, a brief explanation of the reason must be documented in this log.

14.1.2 Overwriting of data files is never allowed.

14.2 The Standard Preparation Logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

14.2.1 The SGS Lot Number must be cross-referenced on the standard vial.

14.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

14.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

14.5 Unused blocks of any form must be X'ed or Z'ed out by the analyst before submitting the data for review.

14.6 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information must also be verified during this review.

15.0 DATA REVIEW AND REPORTING

15.1 Initial and continuing calibration check. Verify that all calibration and continuing calibration criteria have been achieved. If the criteria had not been achieved, corrective action must be performed to bring the system in control before analyzing any samples.

15.1.1 If samples had been analyzed under non-compliant calibration criteria, all sample extracts must be re-analyzed once the system is brought into control.

15.2 Quality Control Data Review. Review all QC data. If QC criteria were not achieved, perform corrective action before proceeding with analysis.

15.2.1 In some situation, corrective action may demand that the entire sample batch be re-extracted and re-analyzed before processing data.

15.3 Chromatogram Review. The chromatogram of each sample is evaluated for target analytes.

15.3.1 Each sample may require the reporting of different target analytes. Review the login to assure that the correct target compounds are identified.

15.3.2 Manual integration of chromatographic peaks must be identified by the analysts. Upon review, the supervisor will initial and date the changes made to the report.

15.4 Transfer to LIMS. Following the initial screen review, transfer the processed data to the LIMS.

15.4.1 Compare the printed values to the original values to verify transfer accuracy.

15.4.2 If transfer errors occurred, the errors must be corrected before the data is re-submitted.

16.0 POLLUTION PREVENTION & WASTE MANAGEMENT

16.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 16.2.

16.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

16.2.1 Non-hazardous aqueous wastes.
16.2.2 Hazardous aqueous wastes
16.2.3 Chlorinated organic solvents
16.2.4 Non-chlorinated organic solvents
16.2.5 Hazardous solid wastes
16.2.6 Non-hazardous solid wastes

Table 1 – Target Compounds by SW846 8270E			
Benzenethiol	4-Bromophenyl phenyl ether	Di-n-octyl phthalate	5-Nitro-o-toluidine
Benzoic Acid	Butyl benzyl phthalate	Diethyl phthalate	Naphthalene
2-Chlorophenol	Benzyl Alcohol	Dimethyl phthalate	Nitrobenzene
4-Chloro-3-methyl phenol	1,1'-Biphenyl	2,3-Dichloroaniline	n-Nitrosodimethylamine

2,4-Dichlorophenol	Butyl Stearate	Decane	4-Nitroquinoline 1-Oxide
2,4-Dimethylphenol	2-Chloronaphthalene	Octadecane	N-Nitroso-di-n-propylamine
2,4-Dinitrophenol	4-Chloroaniline	bis(2-Ethylhexyl)phthalate	N-Nitrosodi-n-butylamine
2,6-Dichlorophenol	Carbazole	Ethyl methanesulfonate	N-Nitrosodiethylamine
4,6-Dinitro-2-methylphenol	Caprolactam	Famphur	N-Nitrosodiphenylamine
Dinoseb	Chlorobenzilate	Fluoranthene	N-Nitrosomethylethylamine
2-Methylphenol	Chrysene	Fluorene	N-Nitrosomorpholine
3&4-Methylphenol	Cumene	Hexachlorobenzene	N-Nitrosopiperidine
2-Nitrophenol	bis(2-Chloroethoxy)methane	Hexachlorobutadiene	N-Nitrosopyrrolidine
4-Nitrophenol	bis(2-Chloroethyl)ether	Hexachlorocyclopentadiene	O,O,O-Triethyl phosphorothioat
Pentachlorophenol	bis(2-Chloroisopropyl)ether	Hexachloroethane	2-Picoline
Phenol	4-Chlorophenyl phenyl ether	Hexachlorophene	Parathion
2,3,4,6-Tetrachlorophenol	1,2-Dichlorobenzene	Hexachloropropene	Pentachloroethane
2,4,5-Trichlorophenol	1,2-Diphenylhydrazine	Indene	Pentachlorobenzene
2,4,6-Trichlorophenol	1,3-Dichlorobenzene	Indeno(1,2,3-cd)pyrene	Pentachloronitrobenzene
2-Acetylaminofluorene	1,4-Dichlorobenzene	Isodrin	Phenacetin
4-Aminobiphenyl	2,4-Dinitrotoluene	Isophorone	Phenanthrene
Acenaphthene	2,6-Dinitrotoluene	Isosafrole	Phorate
Acenaphthylene	3,3'-Dichlorobenzidine	Kepone	Pronamide
Acetophenone	3,3'-Dimethylbenzidine	1-Methylnaphthalene	Pyrene
Aniline	1,4-Dioxane	2-Methylnaphthalene	Pyridine
Anthracene	7,12-Dimethylbenz(a)anthracene	3-Methylcholanthrene	p-Phenylenediamine
Aramite	Dimethylnaphthalenes (total)	4,4'-Methylenebis(2-chloroaniline)	Quinoline
Atrazine	Diallate	Methapyrilene	Safrole
alpha-Terpineol	Dibenz(a,h)acridine	Methyl methanesulfonate	1,2,4,5-Tetrachlorobenzene
A,A-Dimethylphenethylamine	Dibenzo(a,h)anthracene	Methyl parathion	1,2,4-Trichlorobenzene
Benzidine	Dibenzofuran	6-Methyl Chrysene	1,2,3-Trichlorobenzene
Benzaldehyde	Dimethoate	1,4-Naphthoquinone	1,3,5-Trichlorobenzene
Benzo(a)anthracene	Diphenylamine	1-Naphthylamine	Thionazin
Benzo(a)pyrene	Disulfoton	2-Naphthylamine	o-Toluidine
Benzo(b)fluoranthene	m-Dinitrobenzene	2-Nitroaniline	sym-Trinitrobenzene
Benzo(g,h,i)perylene	p-(Dimethylamine) azobenzene	3-Nitroaniline	Tetraethyl dithiopyrophosphate
Benzo(k)fluoranthene	Di-n-butyl phthalate	4-Nitroaniline	

Table 2 – RECOMMENDED OPERATING CONDITIONS: Gas Chromatograph/ Mass Spectrometer	
Injection Type	Splitless
Carrier Gas (linear velocity)	Helium at 30 cm/sec*
Mass range	35-500 AMU
Electron Energy	70 volts (nominal)
Scan time	not to exceed 1 sec. per scan
Injection port temperature	200-300 °C
Source temperature	220-270 °C
Transfer line temperature	250-300 °C
Analyzer temperature	220-250 °C
Injection volume	1 ul (5 ul for LVI)
Gas Chromatograph Temperature Program*	
Initial temperature	40-50 °C*
Time 1	2-4 minutes*
Column temperature rate	8-25 degrees/min*
Final temperature	290-320 °C according to column type*
Total run time	*20-40 minutes*

***** Parameter modification allowed for performance optimization as long as QC criteria are achieved.

Table 2a – SIM Group Parameters		
Group No.	**Retention Time (minutes)**	**Ions**
1	0 – 7.8	150, 64, 93, 82, 152, 99, 63, 128, 112, 42, 95, 88, 58
2	7.8 – 11	150, 128, 225, 142, 172, 152, 129, 223, 141, 171, 122, 127, 227, 115, 170
3	11 – 13.8	172, 152, 166, 182, 334, 266, 176, 153, 165, 330, 284, 264, 174, 154, 77, 332, 286, 268
4	13.8 – 18	266, 179, 202, 122, 268, 212, 203, 284, 178, 213, 244, 286
5	18 – 22	244, 229, 167, 122, 226, 202, 228, 149, 203
6	22 – 34.7	264, 149, 253, 278, 263, 150, 250, 139, 265, 252, 276, 138

Table 3 - DFTPP KEY IONS AND ION ABUNDANCE CRITERIA	
Mass	**Ion Abundance Criteria**
51	30-60 of mass 198
68	<2 % of mass 69
70	<2 % of mass 69
127	40-60 % of mass 198
197	<1 % of mass 198
198	Base peak, 100 % relative abundance
199	5-9 % of mass 198
275	10-30 % of mass 198
365	>1 % of mass 198
441	Present but less than mass 443
442	>40 % of mass 198
443	17-23 % of mass 442

Table 4 – INTERNAL STANDARDS	
Internal Standard (Full Scan)	**Prim/Sec. ions**
1,4-Dichlorobenzene-d4	152 / 150, 115
Naphthalene-d8	136 / 68
Acenaphthene-d10	164 / 162, 160
Phenanthrene-d10	188 / 94, 80
Chrysene-d12	240 / 120, 236
Perylene-d12	264 / 260, 265
Internal Standard (SIM)	**Prim/Sec. ions**
1,2-Dichlorobenzene-d4	152/ 150
1-Methylnaphthalene-d10	150/ 152, 122
Fluorene-d10	174/ 176
Fluoranthene-d10	212/ 213
Benzo(a)pyrene- d12	264/ 263, 265
1,4-Dioxane-d8	96/ 64

Table 6 – Full Scan Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation			
1,4-Dichlorobenzene-d4	**Ions**	**Acenaphthene-d10**	**Ions**
Aniline	(93/66,65)	Acenaphthene	(154/153,152)
Benzaldehyde	(105)	Acenaphthylene	(152/151,153)
Benzenethiol	(110)	1-Chloronaphthalene	(162/127,164)
Benzyl alcohol	(108/79,77)	2-Chloronaphthalene	(162/127,164)
Bis(2-chloroethyl)ether	(93/63,95)	4-Chlorophenylphenyl ether	(204/206,141)
Bis (2-chloroisopropyl)ether	121	Dibenzofuran	(168/139)
2-Chlorophenol	(128/64,130)	Diethyl phthalate	(149/177,150)
Cumene	(105,120)	Dimethyl phthalate	(163/149,164)
Decane	(43)	m-Dinitrobenzene	(168)
1,3-Dichlorobenzene	(146/148,111)	2,4-Dinitrophenol	(184/63,154)
1,4-Dichlorobenzene	(146/148,111)	2,4-Dinitrotoluene	(165/63,89)
1,2-Dichlorobenzene	(146/148,111)	2,6-Dinitrotoluene	(165/63,89)
1,4 Dioxane	(88, 58)	Fluorene	(166/165,167)
Ethyl methanesulfonate	(79/109,97)	Hexachlorocyclopentadiene	(295/237,142)
2-Fluorophenol (SURR.)	(112)	1,4 – Naphthoquinone	(158)
Hexachloroethane	(117/201,199)	1- Naphthylamine	(143/115,116)
Indene	(116)	2- Naphthylamine	(143/115,116)
Methyl methanesulfonate	(80/79,64)	2-Nitroaniline	(65/92,138)
2-Methylphenol	(108/107,79)	3-Nitroaniline	(138/108,92)
4-Methylphenol	(108/107,79)	4-Nitroaniline	(138/108,92)
N-Nitrosodiethylamine	(102)	4-Nitrophenol	(139/109,65)
N-Nitrosodimethylamine	(74/42)	5 Nitro-o-toluidine	(152)
N-Nitroso-di-n-propylamine	(70/101,130)	Pentachlorobenzene	(250/252,248)
N-Nitrosomethyethylamine	(42)	Pentachloronitrobenzene	(237/235,272)
N-Nitrosomorpholine	(56)	Phenacetin	(108/109,179)
N-Nitrosoptrrolidine	(41)	Phorate	(75)
O-Toluidine	(106)	Pronamide	(173/175,145)
Petachloroethane	(167)	1,2,4,5-Tetrachlorobenzene	(216/214,218)
Phenol	(94)	2,3,4,6-Tetrachlorphenol	(232/230,131)
Phenol-d5 (SURR.)	(99)	Tetraethyldithiopyrophosphate	(322)
2-Picoline	(93/66,92)	Thioazin	(143)
Pyridine	(79)	2,4,6-Trichlorophenol	(196/198,200)
		2,4,5-Trichlorophenol	(196/198,200)

Table 6 (cont'd) – Full Scan Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation			
Naphthalene-d8	**Ions**	**Phenanthrene-d10**	**Ions**
A,A-Dimethylphenethylamine	(58)	4-Aminobiphenyl	(169/168,170)
Acetophenone	(105/77,51)	Anthracene	(178/176,179)
Benzoic acid	(184/92,185)	Atrazine	(58)
Bis(2-chloroethoxy)methane	(93/95,123)	4-Bromophenyl phenyl ether	(248/250,141)
Caprolactam	(55)	Carbazole	(167)
4-Chloroaniline	(127)	Diallate	(86)
4-Chloro-methylphenol	(107/144)	Dimethoate	(87)
2,3 Dichloroaniline	(161)	Di-n-Butyl phthalate	(149/150)
2,4-Dichlorophenol	(162/164,98)	4,6-Dinitro-2-methylphenol	(198/51,105)
2,6-Dichlorophenol	(162/164,98)	Dinoseb	(211)
Dimethylnaphthalene	(156)	Diphenylamine	(169/168,167)
2,4-Dimethylphenol	(122/107)	1,2-Diphenylhydrazine	(77/105)
a,a-Dimethyl-phenethylamine	(58/91,42)	Disulfoton	(88)
Hexachlorobutadiene	(225/223,227)	Fluoranthene	(202/101,203)
Hexachloroprene	(213)	2-Fluorobiphenyl (SURR)	(172)
Isophorone	(82/95,138)	Hexachlorobenzene	(284/142,249)
Isosafrole	(127)	Isodrin	(193)
1-Methylnaphthalene	(142)	Methapyriline	(58)
2-Methylnaphthalene	(142/141)	Methyl Parathion	(125)
Naphthalene	(128/129,127)	N-Nitrosodiphenylamine	(169/168,167)
Nitrobenzene	(77/123,65)	4-Nitroquinoline 1-oxide	(190)
Nitrobenzene-d5 (SURR.)	(82)	Octadecane	(57)
N-Nitroso-di-n-butylamine	(84/57/41)	Parathion	(109)
2-Nitrophenol	(139/109,65)	Pentachlorophenol	(266/264,268)
Quinoline	(129)	Phenanthrene	(178/179,176)
N-Nitrosopiperidine	(42/114,55)	Pronamide	(173)
p-Phenylenediamine	(108)	sym- Trinitrobenzene	(213)
O,O,O-Triethylphosphorthioat	(198)	2,4,6 Tribromophenol (SURR)	(330)
Safrole	(162)		
alpha –Terpineol	(128)	**Perylene-d12**	**Ions**
1,2,3-Trichlorobenzene	(180/182,145)	Benzo(b)fluoranthene	(252/125)
1,2,4-Trichlorobenzene	(180/182,145)	Benzo(k)fluoranthene	(252/125)
1,3,5-Trichlorobenzene	(180/182,145)	Benzo(g.h.i)perylene	(276/138,277)
		Benzo(a)pyrene	(252/253,125)
Chrysene-d12	**Ions**	Dibenz(a,j)acridine	(279/280)
2 –Acetylaminofluorene	(181)	Dibenz(a,h)anthracene	(278/139,279)
Aramite	(194)	7,12-Dimethylbenz(a)anthracene	(256/241,257)
Benzidine	(184)	Di-n-Octyl Phthalate	(149)
Benzo(a)anthracene	(228/229/226)	Hexachlorophene	(196)
Bis(2-ethylhexyl)phthalate	(149/167,279)	Indeno(1,2,3-d)pyrene	(276)
Butylbenzyl phthalate	(149/91)	3-Methylchloanthrene	(268/253)
Chlorobenzilate	(251)		
Chrysene	(228/226,229)		
3,3'-Dichlorobenzidine	(252/254,126)		
p-Dimethylaminoazobenzene	(120/225,77)		
3,3 Dimethylbenzidine	(212)		
Famphur	(218)		
Kepone	(272)		
Methyl Chrysene	(242)		
Pyrene	(202/200,203)		
Terphenyl-d14 (SURR.)	(244)		
Table 6a – SIM Semivolatile Internal Standards with Corresponding Analytes Assigned for Quantitation			
1,4-Dichlorobenzene-d4	**Ions**	**Fluoranthene-d10**	**Ions**
2-Fluorophenol (Surr)	(112)	Fluoranthene	202, 101, 203
Phenol-d5 (Surr)	(99)	Pyrene	202, 203

Bis-(2-chloro-ethyl)ether	93, 63, 95	Terphenyl-d14 (Surr)	(244)
Nitrobenzene-d5 (Surr)	(82)	Benzo(a)anthracene	228, 229, 226
		Chrysene	228, 226, 229
1-Methylnaphthalene-d10	**Ions**	Bis(2-ethylhexylphthalate	149, 167, 279
1,4-Dioxane	88, 58		
Naphthalene	128, 129, 127		
Hexachlorobutadiene	225, 223, 227	**Benzo(a) pyrene-d12**	**Ions**
2-Methyl Naphthalene	142, 141, 115	Di-n-octyl phthalate	149, 150, 43
2-Fluorobiphenyl (Surr)	(172)	Benzo(b)fluoranthene	252, 253
		Benzo(k)fluoranthene	252, 125
Fluorene-d10	**Ions**	Benzo(a)pyrene	252, 253, 125
Acenaphthylene	152, 151, 153	Indeno(1,2,3-cd)pyrene	276, 277, 138
Acenaphthene	153, 152, 154	Dibenzo(a,h)anthracene	278, 139, 279
Fluorene	166, 165, 167	Benzo(g,h,i)perylene	276, 138, 277
1,2-Diphenylhydrazine	77, 105, 182		
2,4,6-Tribromophenol (Surr)	(330)	**1,4-Dioxane-d8**	**Ions**
Hexachlorobenzene	284, 286	**1,4-Dioxane**	**88, 58**
Pentachlorophenol	266, 264		
Phenanthrene	178, 179, 176		
Anthracene	178, 176, 179		

Table 7 STANDARD PREPARATION

Table 7A – Intermediate Calibration Standard Solution				
Stock Solution	**Stock Conc., µg/mL**	**Volume Added, µl**	**Final Vol. in $MeCl_2$, mL**	**Final Conc. µg/mL**
Semivolatile Standard Mix # 1	2,000	500	10	100
Semivolatile Standard Mix # 2	2,000	500	10	100
Semivolatile Standard Mix # 4	2,000	500	10	100
Semivolatile Standard Mix # 5	2,000	500	10	100
Semivolatile Standard Mix # 6	2,000	500	10	100
Semivolatile Standard Mix # 7	2,000	500	10	100
PAH Mixture #2	2,000	500	10	100
Semivolatile Standard Mix # 8	2,000	500	10	100
Additional Requested Compound(s) Mix	2,000	500	10	100
Pyridines Mixture	2,000	500	10	100
1,2,3-Trichlorobenzene	1,000	1,000	10	100
1,3,5-Trichlorobenzene	1,000	1,000	10	100
Butyl Stearate	10,000	200	10	200
Pentachlorophenol	1,000	1,000	10	100
B/N Surrogate Standard Mix	5,000	200	10	100
Acid Surrogate Standard Mix	7,500	134	10	100.5

Table 7B – Intermediate Calibration Standard Solution -SIM				
Stock Solution	**Stock Conc., µg/mL**	**Volume Added, µl**	**Final Vol. in $MeCl_2$, mL**	**Final Conc. µg/mL**
Semivolatile Standard Mix # 1	2,000	50	10	10
Semivolatile Standard Mix # 2	2,000	50	10	10
Toxic #2	2,000	50	10	10
PAH Mixture #2	2,000	50	10	10
Semivolatile Standard Mix # 8 (Acids)	2,000	250	10	50
1-Methynaphthalene	1,000	100	10	10
B/N Surrogate Standard Mix	5,000	100	10	50
Acid Surrogate Standard Mix (Full Scan)	7500	67	10	50

Table 7C – Initial Calibration Standards Prep Scheme

Standard Solution	Intermediate Conc., µg/mL	Intermediate added, µl Full Scan	Final Volume in $MeCl_2$, mL	Final Conc., µg/mL – Full Scan
STD 1	100	1,000	1	100
STD 2	100	800	1	80
STD 3	100	500	1	50
STD 4	100	250	1	25
STD 5	100/10 (SIM)	100	1	10
STD 6	100	50	1	5
STD 7	100	20	1	2
STD8	100	10	1	1

Table 7D Initial Preparation Standards Prep Scheme - SIM				
Standard Solution	**Intermediate Conc., µg/mL**	**Intermediate added, µl SIM**	**Final Volume in $MeCl_2$, mL**	**Final Conc., µg/mL – SIM Scan**
STD 1	10/50	500	1	5 BN / 25 Acids
STD 2	10/50	250	1	2.5 BN / 12.5 Ac
STD 3	10/50	100	1	1 BN / 5 Acids
STD 4	1	200	1	0.2 BN / 1 Acids
STD 5	1	100	1	0.1 BN / 0.5 Acids
STD7	0.1	500	1	0.05 BN / 0.25 AC
STD 6	0.1	200	1	0.02 BN / 0.1 AC

Table 7E– ICV -Second Source Calibration Check Standard				
Intermediate	**Intermediate Conc., µg/mL**	**Volume Used, µl (Full/SIM)**	**Final Volume in Acetone, mL**	**Final Conc., µg/mL (Full/SIM)**
Base Neutrals Mixture	100	500/ 50	1	50/ 5
Acid Mixture	100	500/ 50	1	50/ 5

Table 7F Initial Preparation Standards Prep Scheme – 1,4-DioxaneSIM isotopic dilution

Standard Solution	Intermediate Conc., µg/mL	Intermediate added, µl SIM	Final Volume in $MeCl_2$, mL	Final Conc., µg/mL – SIM Scan
STD 1	20	1000	1	20
STD 2	20	500	1	10
STD 3	20	250	1	5
STD 4	5	500	1	2.5
STD 5	5	200	1	1
STD7	1	500	1	0.5
STD 6	1	200	1	0.2
STD 7	1	100	1	0.1
STD 8	0.1	500	1	0.05
STD9	0.1	200	1	0.02

Table 7G – Initial Calibration Standards Prep Scheme - LVI

Standard Solution	Intermediate Conc., µg/mL	Intermediate added, µl Full Scan	Final Volume in $MeCl_2$, mL	Final Conc., µg/mL – Full Scan
STD 1	100	200	1	20
STD 2	80	200	1	16
STD 3	50	200	1	10
STD 4	25	200	1	5
STD 5	10	200	1	2
STD 6	5	200	1	1
STD 7	2	200	1	0.4
STD8	1	200	1	0.2

Table 7H Initial Preparation Standards Prep Scheme – SIM LVI

Standard Solution	Intermediate Conc., µg/mL	Intermediate added, µl SIM	Final Volume in $MeCl_2$, mL	Final Conc., µg/mL – SIM Scan
STD 1	5/25	200	1	1 BN / 5 Acids
STD 2	2.5/12.5	200	1	0.5 BN / 2.5 Acids
STD 3	1/5	200	1	0.2 BN / 1 Acids
STD 4	0.2/1	200	1	0.04 BN / 0.2 Acids
STD 5	0.1/0.5	200	1	0.02 BN / 0.1 Acids
STD7	0.05/0.25	200	1	0.01 BN / 0.05 Acids
STD 6	0.02/0.1	200	1	0.004 BN / 0.02 Acids

Table 8a –Selected Ion Monitoring: Masses and Dwell Times		
Compound	**Mass Ion (m/z)**	**Dwell Time (ms)**
Acenaphthene	153, 152, 154	50
Acenaphthylene	152, 151, 153	50
Anthracene	178, 176, 179	50
Benzo(a)anthracene	228, 229, 226	50
Benzo(a)pyrene	252, 253, 125	50
Benzo(b)fluoranthene	252, 253	50
Benzo(g,h,i)perylene	276, 138, 277	50
Benzo(k)fluoranthene	252, 125	50
Chrysene	228, 226, 229	50
Dibenzo(a,h)anthracene	278, 139, 279	50
Fluoranthene	202, 101, 203	50
Fluorene	166, 165, 167	50
Indeno(1,2,3-cd)pyrene	276, 277, 138	50
Naphthalene	128, 129, 127	50
Phenanthrene	178, 179, 176	50
Pyrene	202, 203	50
2-Methyl Naphthalene	142, 141, 115	50
Bis-(2-chloro-ethyl)ether	93, 63, 95	50
Pentachlorophenol	266, 264	50
Hexachlorobutadiene	225, 223, 227	50
1,2-Diphenylhydrazine	77, 105, 182	50
Bis(2-ethylhexyl)phthalate	149, 167, 279	50
Di-n-octyl phthalate	149, 150, 43	50
Hexachlorobenzene	284, 286	50
2-Fluorophenol	112, 64, 63	50
Phenol-d5	99, 42	50
Nitrobenzene-d5	82, 128	50
2-Fluorobiphenyl	172, 171, 170	50
2,4,6-Tribromophenol	330, 332, 334	50
1,4 Dioxane	88, 58	50
4,6-dinitro-2-methylphenol	198, 51,105	50
Terphenyl-d14	244, 122	50

Table 9 REPORTING LIMITS

Compound	Water	Soil	Compound	Water	Soil
	µg/l	µg/kg		µg/l	µg/kg
Benzoic Acid	20	667	Chlorobenzilate	5	167
2-Chlorophenol	5	167	Chrysene	1	33
4-Chloro-3-methylphenol	5	167	bis(2-Chloroethoxy)methane	2	67
2,4-Dichlorophenol	5	167	bis(2-Chloroethyl)ether	2	67
2,4-Dimethlyphenol	5	167	Bis(2-Chloroisopropyl)ether	2	67
2,4-Dinitrophenol	20	667	4-Chlorophenyl phenyl ether	2	67
4,6-Dinitro-o-cresol	20	667	1,2-Dichlorobenzene	2	67
Dinoseb	5	167	1,3-Dichlorobenzene	2	67
2-Methylphenol	2	67	1,4-Dichlorobenzene	2	67
4-Methylphenol	2	67	2,4-Dinitrotoluene	2	67
2-Nitrophenol	5	167	2,6-Dinitrotoluene	2	67
4-Nitrophenol	10	333	3,3'-Dichlorobenzidine	5	167
Pentachlorophenol	10	333	3,3’-Dimethylbenzidine	5	167
Phenol	2	67	7,12-Dimethylbenz(a)anthracene	5	167
2,3,4,6-Tetrachlorophenol	5	167	Diallate	5	167
2,4,5-Trichlorophenol	5	167	Dibenzo(a,h)anthracene	1	33
2,4,6-Trichlorophenol	5	167	Dibenzofuran	2	67
2-Acetylaminofluorene	5	167	Dimethoate	5	167
4-Aminobiphenyl	5	167	Diphenylamine	5	167
Acenaphthene	1	33	Disulfuton	5	167
Acenaphthylene	1	33	m-Dinitrobenzene	5	167
Acetophenone	5	167	p-(Dimethylamine)azobenzene	5	167
Aniline	2	67	Di-n-butyl phthalate	2	67
Anthracene	1	33	Di-n-octyl phthalate	2	67
Aramite	5	167	Diethyl phthalate	2	67
A,A-Dimethylphenethylamine	5	167	Dimethyl phthalate	2	67
Benzo(a)anthracene	1	33	bis(2-Ethylhexyl)phthalate	2	67
Benzo(a)pyrene	1	33	Ethyl methansulfonate	5	167
Benzo(b)fluoranthene	1	33	Famphur	30	1000
Benzo(g,h,i)perylene	1	33	Fluoranthene	1	33
Benzo (k)fluoranthene	1	33	Fluorene	1	33
4-Bromophenyl phenyl ether	2	67	Hexachlorobenzene	2	67
Butyl benzyl phthalate	2	67	Hexachlorobutadiene	1	33
Benzyl Alcohol	2	67	Hexachlorocyclopentadiene	20	667
2-Chloronaphthalene	2	67	Hexachloroethane	5	167
4-Chloroaniline	5	167	Hexachlorophene	50	1700
Carbazole	1	67	Hexachloropropene	5	167

Table 9 (Cont'd)

Compound	Water	Soil	Compound	Water	Soil
	µg/l	µg/kg		µg/l	µg/kg
Indeno(1,2,3-cd)pyrene	1	33	N-Nitrosomethylethylamine	5	167
Isodrin	5	167	N-Nitrosomorpholine	5	167
Isophorone	2	67	N-Nitrosopiperidine	5	167
Isosafrole	5	167	N-Nitrosopyrrolidine	5	167
Kepone	30	1000	O,O,O Triethylphosphorothioat	5	167
2-Methylnaphthalene	2	667	2-Picoline	5	167
3-Methylcholanthene	5	167	Parathion	5	167
Methapyrilene	5	167	Pentachlorobenzene	5	167
Methyl Methanesulfonate	5	167	Pentachloroethane	5	167
Methyl Parathion	5	167	Pentachloronitrobenzene	5	167
1,4 Naphthoquinone	5	167	Phenacetin	5	167
1-Naphthylamine	5	167	Phenanthrene	1	33
2-Naphthylamine	5	167	Phorate	5	167
2-Nitroaniline	5	167	Pronamide	5	167
3-Nitroaniline	5	167	Pyrene	1	33
4-Nitroaniline	5	167	Pyridine	2	67
5-Nitro-o-toluidine	5	167	p-Phenylenediamine	5	167
Naphthalene	1	33	Safrole	5	167
Nitrobenzene	2	67	1,2,4,5 Tetrachlorobenzene	5	167
n-Nitrosodimethylamine	2	67	1,2,4-Trichlorobenzene	2	67
4-Nitroquinoline-1-Oxide	10	333	Thionazin	5	167
N-Nitroso-di-n-propylamine	2	67	o-Toluidine	5	167
N-Nitrosodi-n-butylamine	5	167	sym-Trinitrobenzene	5	167
N-Nitrosodiethylamine	5	167	Tetraethyl dithiopyrophosphate	5	167
N-Nitrosodiphenylamine	5	167	Quinoline	5	167
Benzenethiol	20	667	Indene	5	167
1,4-Dioxane	1	33			

Table 10 Selected Ion Monitoring Reporting Limits

Compound	Water	Soil	Compound	Water	Soil
	µg/l	µg/kg		µg/l	µg/kg
Pentachlorophenol	0.3	17	Fluoranthene	0.1	3.3
Acenaphthene	0.1	3.3	Fluorene	0.1	3.3
Acenaphthylene	0.1	3.3	Hexachlorobenzene	0.02	3.3
Anthracene	0.1	3.3	Hexachlorobutadiene	0.1	3.3
Benzo(a)anthracene	0.1	3.3	Indeno(1,2,3-cd)pyrene	0.1	3.3
Benzo(a)pyrene	0.1	3.3	2-Methylnaphthalene	0.1	3.3
Benzo(b)fluoranthene	0.1	3.3	Naphthalene	0.1	3.3
Benzo(g,h,i)perylene	0.1	3.3	Phenanthrene	0.1	3.3
Benzo (k)fluoranthene	0.1	3.3	Pyrene	0.1	3.3
Chrysene	0.1	3.3	bis(2-Chloroethyl)ether	0.2	6.6
Dibenzo(a,h)anthracene	0.1	3.3	Bis (2-ethylhexyl) phthalate	0.2	6.6
1,2-Diphenylhydrazine	0.2	6.6	Di-n-octyl phthalate	0.2	6.6
4,6-dinitro-2-methylphenol	0.5	16.7	1,4 Dioxane	0.1	3.3
			1,4 Dioxane by Isotopic Dilution	0.3	

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
9.5.1.1	Added 2 ppm to the SIM LVI curve
10.1.1	Added 2 ppm to the SIM LVI curve

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

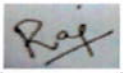
Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
01	03/31/2021	Olga Azarian
02	9/15/2022	Jason Savoie
03	4/13/2023	Jason Savoie
04	8/11/2023	Olga Azarian
05	9/21/2023	Olga Azarian

END OF DOCUMENT

LAB SUPERVISOR: 

QA OFFICER: Jaafar

EFFECTIVE DATE: 02/13/2024

TITLE: DETERMINATION OF PER-AND POLYFLUOROALKYL SUBSTANCES (PFAS) IN AQUEOUS MATRICES BY LIQUID CHROMATOGRAPHY/TANDEM MASS SPECTROMETRY (LC/MS/MS)
REFERENCES: ASTM D8421-22, June 2022
REVISED SECTIONS: NEW SOP

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentrations of select Perfluorinated Alkyl substances (PFAS) in non-potable water utilizing liquid chromatography equipped with a tandem mass spectrometer (MS/MS).

Table 1. The following 40 compounds (with reporting limits, RLs) can be reported by this method:

PFAS Analyte	Acronym	CAS #	RL (ppt)
Perfluorobutanoic acid	PFBA	375-22-4	40
Perfluoropentanoic acid	PFPeA	2706-90-3	20
Perfluorohexanoic acid	PFHxA	307-24-4	10
Perfluoroheptanoic acid	PFHpA	375-85-9	10
Perfluorooctanoic acid	PFOA	335-67-1	10
Perfluorononanoic acid	PFNA	375-95-1	10
Perfluorodecanoic acid	PFDA	335-76-2	10
Perfluoroundecanoic acid	PFUnA	2058-94-8	10
Perfluorododecanoic acid	PFDoA	307-55-1	10
Perfluorotridecanoic acid	PFTriA	72629-94-8	10
Perfluorotetradeconoic acid	PFTeA	376-06-7	10
Perfluorobutane sulfonate	PFBS	29240-43-3	10
Perfluoropentane sulfonate	PFPeS	2706-91-4	10
Perfluorohexane sulfonate	PFHxS	108427-53-8	10
Perfluoroheptane sulfonate	PFHpS	375-92-8	10
Perfluorooctane sulfonate	PFOS	1763-23-1	10

Perfluorononane sulfonate	PFNS	68259-12-1	10
Perfluorodecane sulfonate	PFDS	67906-42-7	10
Perfluorododecanesulfonate	PFDoDS	79780-39-5	10
4:2 Fluorotelomer sulfonate	4:2 FTS	757124-72-4	40
6:2 Fluorotelomer sulfonate	6:2 FTS	27619-97-2	40
8:2 Fluorotelomer sulfonate	8:2 FTS	39108-34-4	40
3:3 Fluorotelomer carboxylate	3:3 FTCA	356-02-5	50
5:3 Fluorotelomer carboxylate	5:3 FTCA	914637-49-3	250
7:3 Fluorotelomer carboxylate	7:3 FTCA	812-70-4	250
N-ethyl perfluorooctanesulfonamido acetic acid	EtFOSAA	2991-50-6	10
N-methyl perfluorooctanesulfonamido acetic acid	MeFOSAA	2355-31-9	10
Perfluorooctane sulfonamide	PFOSA	754-91-6	10
N-Ethyl perfluorooctane sulfonamide	EtFOSA	4151-50-2	10
N-Methyl perfluorooctane sulfonamide	MeFOSA	31506-32-8	10
N-Ethyl perfluorooctane sulfonamidoethanol	EtFOSE	1691-99-2	100
N-Methyl perfluorooctane sulfonamidoethanol	MeFOSE	24448-09-7	100
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6	40
11-chloroicosafluoro-3-oxaundecade-1-sulfonic	11Cl-PF_3ONS	763051-92-9	40
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	9Cl-PF_3ONS	756426-58-1	40
4,8-dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4	40
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6	80
Perfluoro(2-ethoxyethane) sulfonic acid	PFEESA	113507-82-7	80
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1	80
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5	80

2.0 SUMMARY

2.1 This SOP follows the Standard method SW846 3512 (EOP3512) for the dilution of PFAS in non-potable and aqueous film forming (AFFF) impacted water.

2.2 After dilution with methanol (1:1), perfluorinated compounds are separated, detected, and quantitated using an LC/MS/MS. After HPLC separation and ionization, the specific Perfluorinated compound is isolated in the first mass spectrometer and transferred to a

collision cell for fragmentation. The resulting fragments are introduced into the second mass spectrometer where they are detected and quantified.

2.3 Perfluorinated analytes may exist in branched and/or linear form. Fluorotelomer production results in linear isomers only but electrochemical fluorination results in branched and linear isomers. The branched isomers may account for up to 30% of the total analyte. The branched isomer will elute just before the linear isomer.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 The reporting limits (RL) are based on the direct injection procedure and the lowest calibration standard. Reporting limits may vary depending on matrix complications and volumes. Reporting limits for all the PFAS listed for this method are in Table 1.

3.2 The Method Detection Limit (MDL) for each analyte is evaluated using 40 CRF136, Appendix B, Revision 2 procedure, and lab's SOP EQA075.

3.3 Compounds detected at concentrations between the RL, and MDL are quantitated and qualified as estimated values and reported with the appropriate qualifier. Some program or project specifications may require that no values below the RL be reported.

4.0 DEFINITIONS

4.1 Batch: A group of samples that are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.3 Reagent Blank (RB): are prepared and analyzed with all samples and are used to demonstrate that laboratory supplies and preparation and analysis steps do not introduce interferences or PFAS artifacts at levels that would prevent the proper identification and integration of target analytes or bias quantitation, especially near the Lower Limit of Quantitation (LLOQ) or any project-specific concentration levels of interest.

4.4 Field Blank (FB): An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are present in the field environment.

4.5 Holding Time: The maximum times that samples may be held before preparation and/or analysis and still considered valid.

4.6 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the LLOQ. Before the analysis of samples.

4.7 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible or from a manufacturer's batch prepared independently from the batch used for calibration. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.8 Matrix Spike (MS): A sample spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix. One set (MS and MSD) per preparation of 20 or fewer field samples, if sufficient replicate samples are provided).

4.9 Matrix Spike Duplicate (MSD): A replicate sample spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process. One per preparation of 20 or fewer samples.

4.11 Laboratory Control Spike (LCS): The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume, like the MB. The LCS is spiked with the same analytes and at the same concentrations as the MS/MSD, near the midpoint of the initial calibration range, when appropriate, and is taken through all sample preparation steps. One per preparation batch of 20 or fewer samples.

4.12 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.13 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.14 per- and polyfluoroalkyl substances (PFAS), n-synthetic organofluorine chemical compounds with multiple fluorine atoms that include PFOA, PFOS, GenX, and many other chemicals.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Dayton Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of

the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 Methanol is an inhalation hazard. Use in a well-ventilated area.

6.0 COLLECTION, PRESERVATION AND HOLDING TIME

6.1 Collection and Preservation

6.1.1 Samples shall be collected in 60 - 250mL polypropylene (PP) or polyethylene (HDPE) bottles. Caps must not have Teflon liners. Various studies have shown that HDPE is preferable to PP for this analysis.

6.1.2 The samples must be refrigerated at ≤ 6°C from the time of receipt until extraction but must not be frozen.

6.1.3 Water and containers used for equipment blanks or field blanks must be tested prior to use. For smaller sampling events DI water will be provided in the same type of bottle used for sample collection. The DI water and container blanks must be free of any analytes of interest or interferences at ½ the required RL to be acceptable.

6.1.4 A field blank must be collected with each set of samples. Each field blank consists of 4 bottles. Two bottles are filled with DI water at the lab and the other two bottles are empty. At the sampling site the sampler must open then two empty bottles and transfer the DI water from the full bottles into them. Cap the bottles, label as field blanks, and return them to the laboratory along with the samples for analysis.

6.2 Holding Time

6.2.1 Samples must be analyzed within 28 days of collection.

7.0 APPARATUS

7.1 HPLC – Agilent Technologies 1290 Infinity II or comparable HPLC equipped with an autosampler, pump, and column compartment.

7.2 MS/MS – Agilent Technologies 6470B/6495A

7.3 LC/MS/MS must be capable of negative ion electrospray ionization near the required flow rate of the HPLC Column. The system must be capable of performing MS/MS to produce unique precursor and product ions for the PFAA method analytes within the specified retention time segments. A minimum of 10 scans across each peak is required to ensure adequate precision.

7.4 Data System – Agilent Technologies MassHunter B.08.0x. The computer system interfaced to the HPLC/MS/MS must allow for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program. The software must allow for the viewing of the specific MS/MS Spectra acquired over the analytical run. Comparisons can then be made between spectra from standards and samples. Data is archived to a backup server for long-term storage.

7.5 Analytical Column – Agilent ZORBAX RRHD Stable Bond C18, 100 x 2.1 mm, 1.8 µm (p/n – 858700-902)

7.6 Isolator Column - Agilent ZORBAX RRHD Eclipse Plus C18, 50 x 3 mm, 1.8 µm) p/n – 959757-302.

7.7 Disposable polyethylene transfer pipettes

7.8 15ml Centrifuge tubes

7.9 Polyethylene screw cap and autosampler vials

7.10 Volumetric Pipettors and volumetric “plasticware” for dilutions of standards and extracts.

7.11 250mL HDPE bottles – shown to be PFC-free.

7.12 Agilent’s 0.2 µm syringe filter (p/n 5190-5110) demonstrated contaminant-free below ½ MRL.

8.0 REAGENTS

8.1 Water – LCMS grade or equivalent

8.2 Water with 0.1% acetic acid: Add 1 mL of acetic acid to 1.0 L of LCMS grade Water.

8.3 Methanol (CAS #67-56-1)- LCMS grade or equivalent

8.4 0.1 % acetic acid in Methanol: Add 1 mL of acetic acid to 1.0 L of LCMS grade methanol.

8.5 Acetic acid – ACS grade or equivalent

8.6 Perfluorinated Alkyl substances stock standards – Traceable to Certificate of Analysis.

8.7 Mass labeled – Non-Extracted Internal Standards

$^{13}C_3$-PFBA	$^{13}C_4$-PFOA	$^{13}C_2$-PFDA	$^{13}C_4$-PFOS
$^{13}C_2$-PFHxA	$^{13}C_5$-PFNA	$^{18}O_2$-PFHxS	

8.8 Mass labeled – Extracted Internal Standards

$^{13}C_4$-PFBA	$^{13}C_9$-PFNA	$^{13}C_3$-PFBS	$^{13}C_2$-8:2 FTS	D_5-NEtFOSAA
$^{13}C_5$-PFPeA	$^{13}C_6$-PFDA	$^{13}C_3$-PFHxS	$^{13}C_8$-PFOSA	D_7-NMeFOSE
$^{13}C_5$-PFHxA	$^{13}C_7$-PFUnA	$^{13}C_8$-PFOS	D_3-NMeFOSA	D_9-NEtFOSE
$^{13}C_4$-PFHpA	$^{13}C_2$-PFDoA	$^{13}C_2$-4:2 FTS	D_5-NEtFOSA	$^{13}C_3$-HFPO-DA
$^{13}C_8$-PFOA	$^{13}C_2$-PFTeDA	$^{13}C_2$-6:2 FTS	D_3-NMeFOSAA	

8.8 pH paper (pH range 1–14).

8.9 Acetonitrile (CAS #75-05-8).

8.10 Ammonium acetate (CAS #631-61-8).

8.11 2-Propanol (isopropyl alcohol, CAS#67-63-0)

9.0 INTERFERENCES

9.1 Data from all blanks, samples, and spikes must be evaluated for interferences. Method interferences may be caused by contaminants in solvents, reagents, or glassware. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) or Teflon products, HPLC solvent lines, methanol, aluminum foil, SPE transfer lines, bottle caps, etc. All these materials must be demonstrated to be free from interference.

9.2 During method development the following compounds showed a potential for loss either during standard preparation (resulting in low bias to calibration standards and high recoveries for samples) or during sample preparation (resulting in low recoveries). Extra care should be taken to ensure that the composition of the stock and intermediate standards (those above the high calibration standard) maintain a high enough proportion of organic cosolvent to limit loss from the solution. Subsampling from aqueous sample containers prior to adding sufficient organic solvent will also result in a loss of these and potentially other compounds from the solution, the extent of which will be container dependent.

Perfluoroundecanoic acid (PFUnDA)
Perfluorododecanoic acid (PFDoDA)
Perfluorotridecanoic acid (PFTrDA)
Perfluorotetradecanoic acid (PFTeDA)
N-methylperfluoro-1-octanesulfonamidoacetic acid (N-MeFOSAA)
N-ethylperfluoro-1-octanesulfonamidoacetic acid (N-EtFOSAA)

9.3 The following compounds lack or have a low abundance of secondary product ions, and interferences may make qualitative identification more difficult. Perfluorobutanoic acid (PFBA). Perfluoropentanoic acid (PFPeA) Perfluorohexanoic acid (PFHxA) Perfluoro-1-octanesulfonamide (PFOSA).

9.4 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the water. Humic and/or fulvic material can be present in the matrix and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries. Total organic carbon (TOC) is a good indicator of the humic content of the sample.

9.5 Background contamination must be carefully evaluated and managed to a level is acceptable for the project-specific data application.

10.0 PROCEDURE

10.1 Standards Preparation

10.1.1 Standards Preparation. Standards are prepared from commercially available certified NIST or reference standards. All standards must be logged in the HPLC Standards Logbook. All standards shall be traceable to their original source. The standards must be stored at ≤ 6°C, or as recommended by the manufacturer.

10.1.2 Stock Standard Solutions. Stock standards are available from some commercial vendors. All vendors must supply a "Certificate of Analysis" with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor's expiration date. Once opened, the hold time is reduced to one year or the vendor's expiration date (whichever is shorter).

10.1.3 Intermediate Standard Solutions. Intermediate standards are prepared by quantitative dilution of the stock standard. The hold time for intermediate standards is six months or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparisons to other standards indicate analyte degradation or concentration changes. Intermediate standards must be stored in polyethylene vials.

10.1.4 Calibration Standards. Calibration standards for Perfluorinated analytes are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. The low standard is at a concentration at or below the RL and the remaining standards define the working range of the detector. Calibration standards must be stored in polyethylene vials.

Note: Perfluorinated analytes may exist in branched and linear form. If a branched form is commercially available, then the calibration standards must contain the branched and linear form. PFHxS, PFOS, MeFOSAA and EtFOSAA are currently available in mixes of branched and linear isomers.

10.1.5 Calibration standard concentrations must be verified by the analysis of an initial calibration verification (ICV) standard prepared by a different manufacturer.

10.2 HPLC/MS/MS Conditions

10.2.1 HPLC Conditions

35 µL autosampler injection
Autosampler temperature – 5 to 10 °C
Column temperature – 50.0 °C
Flow – 0.4 mL/min

10.2.2 **Gradient Program.**

Time (min)	**0.1% acetic acid in Water**	**0.1% acetic acid in Methanol**
0	98%	2%
0.20	90%	10%
4.00	50%	50%

7.00	25%	75%
9.00	10%	90%
10.00	5%	95%
10.30	0%	100%
12.5	0%	100%
12.6	98%	2%
14.5	98%	2%

10.2.2 MS/MS Conditions. Fragmentation voltages and collision energies are optimized for each analyte and are stored in the instrument method. Precursor ions and transition masses are listed in Table 2.

Parameter	Value	Parameter	Value
Gas Temp (C)	250	Sheath Gas Flow (L/min)	10
Gas Flow (L/min)	10	Capillary (V)	4000
Nebulizer (psi)	50	V Charging	500
Sheath Gas Heater	300	Ionization Mode	Neg ESI

LC/MS/MS conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

10.3 Sample Preparation. Samples are prepared using the SW846 3512 method (EOP3512-00).

10.4 HPLC/MS/MS Analysis.

Instrument calibration consists of four major sections:

Mass Tuning and Calibration
Transition Window Selection
Initial Calibration Procedures
Continuing Calibration Verification

10.4.1 Mass Tuning & Calibration and Transition Window Selection.

10.4.1.1 Before samples can be run, the LC/MS/MS system must be mass-tuned and calibrated.

10.4.1.2 The instrument must be full hardware tuned per the manufacturer's instructions after any maintenance is performed. A check tune must be performed weekly or prior to analyzing a new calibration curve. The instrument must have a valid mass calibration prior to any sample analysis. The mass calibration must be updated as needed. (i.e. QC failures, ion masses showing large deviations from known masses, or major instrument maintenance is performed).

10.4.1.3 Verify the instrument tune and mass calibration by analyzing a mid-point Perfluorinated compound standard. This may be done using the daily CCV. The ions must be within ± 0.5 *m/z* of the expected mass.

10.4.1.4 The mid-point standard is also used to check the analyte retention times. These retention times are used to update the transition windows. The windows must be wide enough to ensure that the branched and linear isomer for PFHxS, PFOS, MeFOSAA and EtFOSAA are completely within the transition window. The branched isomer will elute just prior to the linear isomer. If they are partially cut off, adjust the retention time of the linear isomer or the width of the transition window. Use a similar size window for the other analytes that do not have a branched standard. Later eluting peaks are broader and require a slightly wider transition window.

10.4.2 Initial Calibration Procedures.

Before samples can be run, the LC/MS/MS system must be calibrated.

10.4.2.1 Internal Standard Calibration.

A minimum 5-point calibration curve is created for the Perfluorinated compounds and surrogates using an internal standard technique. SGS - Dayton routinely performs a 7 or 8-point calibration to maximize the calibration range.

The calibration standards for PFHxS, PFOS, MeFOSAA and EtFOSAA must consist of both branched and linear isomers. The branched isomer elutes just prior to the linear isomer. PFCs are currently being reported as the sum of the branched and linear isomers so both peaks must be integrated.

Use the LC/MS/MS data system to generate a linear regression or quadratic calibration curve for each of the analytes. This curve **must** be forced through zero and may be concentration weighted. Forcing zero allows for a better estimate of the background levels of method analytes.

The correlation coefficient (r^2) must be ≥0.99 or (r) ≥0.995. If average calibration factor is used, the RSD must be ≤20%. Additionally, each calibration point for each analyte must be recalculated against the current initial calibration. The recovery of each analyte must be within 70%-130% at higher concentrations and 50%-150% at the LLOQ. If more than 10% of the target compounds do not meet the ICAL acceptance criteria, then the system is considered unacceptable. The source of problem must be corrected, and the instrument must be re-calibrated before analyzing any samples.

10.4.2.2 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV must be prepared from a second source at a mid-range concentration. If second source standards are not available, same calibration standard can be prepared as ICV by another analyst.

NOTE: Second source standards may consist of linear isomers only.

The %D for the compound of interest must be ≤ 30%. If the ICV does not meet these criteria, a second standard must be prepared. If the ICV still does not meet criteria, analyze an ICV prepared from a third source (if available). If this ICV meets criteria, proceed with sample analysis. If the ICV still does not meet criteria, determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

10.4.2.3 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Generally internal calibration methods utilize relative retentions times. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window must be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over a period of time. The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ±3 times the standard deviation of the mean absolute retention time or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks internal standards and surrogates are used to correct for run-to-run variations in retention times due to temperature, flow, or injector

fluctuations. HPLC retention times tend to shift more than GC retention times.

The retention time windows must be used as a guide for identifying compounds; however, the experience of the analyst must weigh heavily in the interpretation of the chromatograms. The analyst must monitor the retention times of known peaks (internal standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential "hits". The analyst will then review these "hits" and determine if the retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

10.4.3 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for the PFAS are prepared at various concentrations. The opening CCV must be prepared at or below the Reporting Limit in order to verify instrument sensitivity.

A CCV must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally, the mid-point CCV must be analyzed after every 10 samples.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| for each analyte of interest must be ≤ 30% in the mid-point CCV and ≤ 50% in the low-level CCV. The |%D| for each surrogate must be ≤ 30% in any CCV.

If the first continuing calibration verification does not meet the criteria, a second standard may be injected. If the second standard does not meet the criteria, the system must be recalibrated.

If the |%D| is outside the control limits, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported, i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed, or the data must be qualified.

10.4.4 Sample Analysis

10.4.4.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Initial Calibration	Whenever needed
ICV - Second Source Calibration Check Standard	Following initial calibration
CCV-Calibration Verification Standard	Every 12 hours
Method Blank	Every 12 hours
Blank Spike	One per analytical batch
Field samples	20 samples or less
Matrix Spike	One per analytical batch
Matrix Spike Duplicate or Sample DUP (depends on sample volume)	One per analytical batch
Closing CCV	At the end of the sequence

10.4.4.2 Two hundred microliters of internal standard solution is added to 10 mL sample (after dilution) in a 15-mL centrifuge tube and filtered through syringe filter, from which 500uL is transferred to the autosampler vial with a pipette.

10.4.4.3 30 – 35 µL microliters (same amount as standards) of sample is injected into the HPLC by the autosampler. The data system then records the resultant peak responses and retention times.

10.4.4.4 Tentative identification of an analyte occurs when the peak from the sample extract falls within the retention time window of the target compound.

10.4.4.5 Positive identification is confirmed by comparing the mass spectra in the sample to the mass spectra of the standards.

10.4.4.6 Some of the PFAAs may have multiple chromatographic peaks due to the presence of linear and branched isomers. This is prevalent in PFHxS, PFOS, MeFOSAA and EtFOSAA. The areas of all the linear and branched isomers peaks must be included and the concentrations reported as a total for each of these analytes.

NOTE: The branched isomers must be included in the quantitation even if the calibration is based on just the linear isomer.

10.4.4.7 If the compound identification does not confirm, then the result must be reported as ND or “U”.

10.4.4.8 If the analyte response exceeds the linear range of the system, the sample must be diluted and reanalyzed. It is recommended that the sample be diluted so that the response falls into the middle of the calibration curve.

11.0 INSTRUMENT MAINTENANCE AND TROUBLESHOOTING

11.1 All instrument maintenance must be documented in the appropriate "Instrument Repair and Maintenance" log. The log will include such items as a problem, action taken, correction verification, date, and analyst.

11.2 Routine maintenance includes:

Fill Mobile Phase Reservoirs	as needed
Empty Waste Reservoir	as needed
Leak and pressure check	as needed
Column back flush	high back pressure
Replace column	as needed
Eluent bottle filter replacement	as needed
Clean Source	as needed
Flush Nebulizer	as needed
Clean Skimmer	as needed
Run Check tune	weekly

11.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Senior reviewer responsible for the instrument must complete the log if the repair technician does not.

11.4 PC and software changes must be documented in the "Instrument Repair and Maintenance" log. Software changes may require additional validation.

12.0 QUALITY ASSURANCE / QUALITY CONTROL

12.1 Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

12.2 Blank spike, blank spike duplicate, matrix spike, and matrix spike duplicate samples are compared to method-defined control limits. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

12.3 Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD). All control limits are updated annually and are listed in the LIMS.

12.4 Internal Standards

12.4.1 The compounds mentioned in section 8.8 are used as internal standards for this method.

12.4.2 The response of the internal standard in all subsequent runs must be 70-130% of the internal standard response in the most recent CCV and ±50% of the internal standard average response from the initial calibration.

12.4.3 If the internal standard responses are not within limits, the following are required.

12.4.3.1 Check to be sure that there are no errors in calculations, integrations, or internal standards solutions. If errors are found, recalculate the data accordingly.

12.4.3.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

12.4.3.3 If no problem is found, prepare a second aliquot of extract and reanalyze the sample.

12.4.3.4 If upon reanalysis, the responses are still not within limits, the problem is considered matrix interference.

12.4.3.5 The sample may need to be reported from the dilution or the results qualified.

12.5 Method Blank. The method blank is either de-ionized or LCMS grade water to which the surrogate standard has been added. The method blank is then taken through all procedures along with the other samples to determine any contamination from reagents, glassware, or high-level samples. The method blank must be free of any analytes of interest or interferences at 1/3 the required reporting level to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples or qualifying the results with the appropriate qualifier.

12.5.1 If the MB is contaminated but the samples are non-detect, then the source of contamination must be investigated and documented. The sample results can be reported without qualification.

12.5.2 If the MB is contaminated but the sample results are >3 times the contamination level, the source of the contamination must be investigated and documented. The sample results may be reported with the appropriate qualifier. This must be approved by the department supervisor.

12.5.3 If the MB is contaminated but the results of the sample are <3 times the contamination level, the source of the contamination must be investigated and documented. The samples must be re-prepared and reanalyzed for confirmation. If there is insufficient sample, or if the sample is prepared beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

12.6 Blank Spike. The blank spike is either de-ionized or LCMS grade water to which the surrogate standard and spike standard have been added. The blank spike is then taken through all procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

12.6.1 Preliminary acceptance limits for the BS are 60-140% and ≤30 RPD for BS duplicate. Statistically derived control limits may be used once enough data is collected.

12.6.2 If the blank spike recoveries are not within the established control limits, the following are required.

12.6.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

12.6.2.2 Check instrument performance. It may be necessary to re-vial and reinject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

12.6.2.3 If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable.

12.6.2.4 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples or qualifying the results as estimated.

12.6.2.5 If there is insufficient sample to prepare, or if the sample is prepared beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

12.6.3 Low-Level Blank Spike (MRL Check sample) - For each batch of 20 or fewer samples, or within a 24-hour analysis window, analyze an MRL check sample. Low-level blank spike is HPLC grade water to which the spike standard has been added at no more than 2 times the LLOQ. The low-level blank spike is then taken through all procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery limits for the MRL are 50 to 150 %. As additional analytes are added to this method, the recoveries will need to be carefully evaluated.

12.6.4 If the low-level blank spike recoveries are not within the control limits, the following are required. Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

12.6.5 Check instrument performance. It may be necessary to re-vial and re-inject the extract to verify performance. If an instrument performance problem is identified, correct the problem, and reanalyze the sample.

12.6.6 If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable.

12.6.7 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

12.6.8 If there is insufficient sample to re-extract, or if the sample is re- extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

12.7 Matrix Spike and Matrix Spike Duplicate. Matrix spike and spike duplicates are replicate sample aliquots to which the spike standard has been added. The matrix spike and spike duplicate are then taken through all procedures along with the other samples to monitor the precision and accuracy of the procedure. The percent recovery for each analyte is calculated as follows:

$$\% \text{ Recovery} = [(\text{Spike Amount} - \text{Sample Amount}) / \text{Amount Spiked}] \times 100$$

12.7.1 Preliminary acceptance limits for the MS/MSD are 60-140% and ≤30 RPD for MS duplicate. Statistically derived control limits may be used once enough data is collected.

12.7.2 If the matrix spike recoveries are not within the established control limits, the following are required.

12.7.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

12.7.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

12.7.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample-related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are indications of the sample matrix effects.

12.7.3 Precision. Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound. The RPD for each

Perfluorinated compound must be less than 30%. Statistically derived control limits may be used once enough data is collected.

12.7.4 If the RPDs fall outside of the established control limits, the MS and MSD must be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

$$RPD = [| MS\ Result - MSD\ Result | / Average\ Result] \times 100$$

13.0 CALCULATIONS

13.1 The concentration of each Perfluorinated compound in the original sample is calculated as follows:

$$Water\ (ug/l) = (CONC_{inst}) \times (V_F / V_I) \times DF$$

$CONC_{inst}$	=	Instrument concentration calculated from the initial Calibration using curve fit
DF	=	Dilution Factor
V_F	=	Volume of final extract (ml)
V_I	=	Volume of sample extracted (ml)

14.0 DOCUMENTATION

14.1 The Analytical Logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

14.1.1 If samples require reanalysis, a brief explanation of the reason must be documented in this log.

14.1.2 Overwriting of data files is never allowed.

14.2 The Standard Preparation Logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

14.2.1 The SGS Dayton Lot Number must be cross-referenced on the standard vial.

14.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

14.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

14.5 Unused blocks of any form must be X'ed or Z'ed out by the analyst before submitting the data for review.

14.6 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information must also be verified during this review.

15.0 DATA REVIEW AND REPORTING

15.1 Initial and continuing calibration check. Verify that all calibration and continuing calibration criteria have been achieved. If the criteria had not been achieved, corrective action must be performed to bring the system in control before analyzing any samples.

15.1.1 If samples had been analyzed under non-compliant calibration criteria, all sample extracts must be re-analyzed once the system is brought into control.

15.2 Quality Control Data Review. Review all QC data. If QC criteria were not achieved, perform corrective action before proceeding with the analysis.

15.2.1 In some situations, corrective action may demand that the entire sample batch be re-extracted and re-analyzed before processing data.

15.3 Chromatogram Review. The chromatogram of each sample is evaluated for target analytes.

15.3.1 Each sample may require the reporting of different target analytes. Review the login to ensure that the correct target compounds are identified.

15.3.2 Manual integration of chromatographic peaks must be identified by the analysts. Upon review, the supervisor will initial and date the changes made to the report.

15.4 Transfer to LIMS. Following the initial screen review, transfer the processed data to the LIMS.

15.4.1 Compare the printed values to the original values to verify transfer accuracy.

15.4.2 If transfer errors occurred, the errors must be corrected before the data is re-submitted.

16.0 POLLUTION PREVENTION & WASTE MANAGEMENT

16.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 16.2.

16.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

16.2.1 Non-hazardous aqueous wastes

16.2.2 Hazardous aqueous wastes

16.2.3 Chlorinated organic solvents.

16.2.4 Non-chlorinated organic solvents

16.2.5 Hazardous solid wastes

16.2.6 Non-hazardous solid wastes

TABLE 2: Precursor and Primary Transition Masses

Analyte	Type	RT	Primary Transition	Secondary Transition	Reference Compound
13C3-PFBA	NIS	3.34	216.0 -> 172.0		
13C4-PFBA	EIS	3.34	216.8 -> 171.9		13C3-PFBA
PFBA	Target	3.34	212.8 -> 168.9		13C4-PFBA
PFMPA	Target	3.99	229.0 -> 84.9		13C5-PFPeA
3:3FTCA	Target	4.32	241.0 -> 177.0	241.0 -> 117.0	13C5-PFPeA
13C5-PFPeA	EIS	4.93	268.3 -> 223.0		13C2-PFHxA
PFPeA	Target	4.93	263.0 -> 219.0		13C5-PFPeA
PFMBA	Target	5.38	279.0 -> 85.1		13C5-PFPeA
13C2-4:2FTS	EIS	5.85	329.1 -> 80.9		18O2-PFHxS
4:2FTS	Target	5.85	327.1 -> 307.0	327.1 -> 80.9	13C5-PFHxA
NFDHA	Target	6.08	295.0 -> 201.0	295.0 -> 84.9	13C5-PFHxA
13C3-PFBS	EIS	6.15	302.1 -> 79.9		18O2-PFHxS
PFBS	Target	6.15	298.7 -> 79.9	298.7 -> 98.8	13C3-PFBS
13C2-PFHxA	NIS	6.20	315.1 -> 270.0		
13C5-PFHxA	EIS	6.20	318.0 -> 273.0		13C2-PFHxA
PFHxA	Target	6.20	313.0 -> 269.0	313.0 -> 118.9	13C5-PFHxA
13C3-HFPO-DA	EIS	6.59	286.9 -> 168.9		13C2-PFHxA
HFPO-DA	Target	6.59	284.9 -> 168.9	284.9 -> 184.9	13C3-HFPO-DA
PFEESA	Target	6.71	314.8 -> 134.9	314.8 -> 82.9	13C5-PFHxA
5:3FTCA	Target	6.82	341.0 -> 237.1	341.0 -> 217.0	13C5-PFHxA
13C4-PFHpA	EIS	7.14	367.1 -> 322.0		13C2-PFHxA
PFHpA	Target	7.14	363.1 -> 319.0	363.1-> 169.0	13C4-PFHpA

PFPeS	Target	7.22	349.1 -> 79.9	349.1 -> 98.9	13C3-PFHxS
ADONA	Target	7.40	376.8 -> 250.9	376.8 -> 84.8	13C3-HFPO-DA
13C2-6:2FTS	EIS	7.56	429.1 -> 80.9		18O2-PFHxS
6:2FTS	Target	7.56	427.1 -> 407.0	427.1 -> 80.9	13C4-PFOA
13C4-PFOA	NIS	7.81	417.1 -> 172.0	417.1 -> 372.0	
13C8-PFOA	EIS	7.81	421.0 -> 376.0		13C4-PFOA
PFOA	Target	7.81	413.0 -> 369.0	413.0 -> 169.0	13C8-PFOA
PFHxS	Target	7.96	398.9 -> 79.9	398.9 -> 98.9	13C3-PFHxS
18O2-PFHxS	NIS	7.97	403.0 -> 83.9		
13C3-PFHxS	EIS	7.97	402.1 -> 79.9		18O2-PFHxS
7:3FTCA	Target	8.27	441.0 -> 316.9	441.0 -> 336.9	13C5-PFHxA
13C5-PFNA	NIS	8.40	468.0 -> 427.0		
13C9-PFNA	EIS	8.40	472.1 -> 427.0		13C5-PFNA
PFNA	Target	8.40	463.0 -> 419.0	463.0 -> 219.0	13C9-PFNA

TABLE 2: Precursor and Primary Transition Masses

Analyte	Type	RT	Primary Transition	Secondary Transition	Reference Compound
PFHpS	Target	8.58	449.0 -> 79.9	449.0 -> 98.8	13C8-PFOS
13C2-8:2FTS	EIS	8.69	529.1 -> 80.9		18O2-PFHxS
8:2FTS	Target	8.70	527.1 -> 507.0	527.1 -> 80.8	13C2-PFDA
13C2-PFDA	NIS	8.95	515.1 -> 470.1		
13C6-PFDA	EIS	8.95	519.1 -> 474.1		13C2-PFDA
PFDA	Target	8.95	512.9 -> 469.0	512.9 -> 219.0	13C6-PFDA
d3-MeFOSAA	EIS	8.97	573.2 -> 419.0		13C4-PFOS
MeFOSAA	Target	8.97	570.1 -> 419.0	570.1 -> 483.0	d3-MeFOSAA
13C4-PFOS	NIS	9.14	503.8 -> 79.9		
13C8-PFOS	EIS	9.13	507.1 -> 79.9		13C4-PFOS
PFOS	Target	9.14	498.9 -> 79.9	498.9 -> 98.8	13C8-PFOS
d5-EtFOSAA	EIS	9.19	589.2 -> 419.0		13C4-PFOS
EtFOSAA	Target	9.20	584.2 -> 419.1	584.2 -> 526.0	d5-EtFOSAA
13C7-PFUnDA	EIS	9.44	570.0 -> 525.1		13C2-PFDA
PFUnDA	Target	9.44	563.1 -> 519.0	563.1 -> 269.1	13C7-PFUnDA
9Cl-PF3ONS	Target	9.49	530.8 -> 351.0	532.8 -> 353.0	13C3-HFPO-DA
PFNS	Target	9.63	548.8 -> 79.9	548.8 -> 98.8	13C8-PFOS
13C2-PFDoDA	EIS	9.87	615.1 -> 570.0		13C2-PFDA
PFDoDA	Target	9.87	613.1 -> 569.0	613.1 -> 319.0	13C2-PFDoDA
PFDS	Target	10.05	599.0 -> 79.9	599.0 -> 98.8	13C8-PFOS
13C8-FOSA	EIS	10.23	506.1 -> 77.8		13C4-PFOS

FOSA	Target	10.23	498.1 -> 77.9	498.1 -> 478.0	13C8-FOSA
PFTrDA	Target	10.26	663.0 -> 619.0	663.0 -> 168.9	13C2-PFDoDA
11Cl-PF3OUdS	Target	10.32	630.9 -> 451.0	632.9 -> 453.0	13C3-HFPO- DA
13C2-PFTeDA	EIS	10.60	715.1 -> 670.0		13C2-PFDA
PFTeDA	Target	10.60	713.1 -> 669.0	713.1 -> 168.9	13C2-PFTeDA
PFDoDS	Target	10.75	699.1 -> 79.9	699.1 -> 98.8	13C8-PFOS
d7-MeFOSE	EIS	11.21	623.1 -> 58.9		13C4-PFOS
MeFOSE	Target	11.22	616.1 -> 58.9		d7-MeFOSE
d3-MeFOSA	EIS	11.30	515.0 -> 219.0		13C4-PFOS
MeFOSA	Target	11.30	512.0 -> 219.0	512.0 -> 169.0	d3-MeFOSA
d9-EtFOSE	EIS	11.45	639.1 -> 58.9		13C4-PFOS
EtFOSE	Target	11.46	630.0 -> 58.9		d9-EtFOSE
d5-EtFOSA	EIS	11.53	531.1 -> 219.0		13C4-PFOS
EtFOSA	Target	11.53	526.0 -> 219.0	526.0 -> 169.0	d5-EtFOSA

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only) :

Section / Subsection	Detailed description of what was revised
All	New SOP

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
00	01/24/2024	Raj Singh

END OF DOCUMENT

LAB SUPERVISOR: Raj

QA OFFICER: Olya Y. Agranovich

EFFECTIVE DATE: 2-22-2024

TITLE: STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF PER- AND POLYFLUORINATED ALKYL SUBSTANCES FROM SOIL SAMPLES FOR LC/MS/MS ANALYSIS

REFERENCES: EPA 1633, January 2024
REVISED SECTIONS: references, 10.24

1.0 SCOPE AND APPLICATION

This procedure is applicable to soil samples submitted for Per fluorinated Alkyl Substances (PFAS) analysis by LC/MS/MS using an Isotope Dilution technique.

2.0 SUMMARY OF METHOD

Soil samples are serially extracted with basic methanol using a shaker table, cleaned with graphitized carbon, concentrated, and back extracted utilizing a solid phase extraction cartridge. The cartridge is eluted with basic methanol, the volume is adjusted to 5.0ml, and stored in centrifuge tube.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 See analytical SOP-EMS1633

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours whichever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Low Level Blank Spike (LLBS): An analyte-free matrix spiked with a known amount of analyte(s) at 2x LLOQ, processed simultaneously with the samples through all the steps of the analytical procedure. Low-Level Blank Spike Recoveries are used to document

laboratory performance at the LLOQ for a given method. This may also be called a Low Level Laboratory Control Sample (LLLCS) or the Low-Level Ongoing Precision And Recovery Standard (LLOPR).

4.4 Extracted Internal Standards (EIS): A standard containing isotopically labelled versions of the native target analytes. These isotopes are usually labelled with C13, d2, or O18 atoms. Isotope Dilution Standards are used to measure the extraction efficiency and to correct the concentrations of the native analytes based on the recovery of their isotopically labelled analogs.

4.5 Field Blank (FB): An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are present in the field environment.

4.6 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and are still considered valid.

4.7 Matrix Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.9 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Laboratory Safety Manual which includes the use of Safety glasses, gloves and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the SDS prior to using any new reagents or solvents.

5.3 Methanol is an inhalation hazard. Use in well ventilated area.

5.4 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets is available to all personnel involved in these analyses.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIME

6.1 Preservation

6.1.1 Samples shall be collected in wide mouth 4oz HDPE jars fitted with a HDPE or Polypropylene screw cap. Alternate size HDPE bottles may be used depending on project requirements. **Glass bottles with Teflon lined caps can NOT be used.**

6.1.2 The samples must be chilled to ≤6°C from the time of collection until arrival at the laboratory. The samples must be refrigerated at ≤ 6°C or frozen at ≤ -20°C from the time of receipt until extraction.

6.1.3 The extracts should be stored at ≤6°C. They must be allowed to come to room temperature prior to analysis. All extracts should be vortexed just prior to transfer to the autosampler vials.

6.2 Holding Time

6.2.1 Soil samples must be extracted and analyzed within 28 days of collection if stored at ≤6°C or within 90 days of collection if stored at ≤ -20°C. The Date/Time that the extraction is started and completed must be recorded on the prep sheet.

7.0 APPARATUS AND MATERIALS

7.1 Solid-phase cartridge extraction system – suitable for use with extraction cartridges

7.2 Vacuum pump

7.3 Vacuum Flasks – or equivalent

7.4 SPE reservoirs – various sizes

7.5 SPE cartridges – Weak Anion Exchange – must have pKa of >8 and 150mg bedsize

7.6 Mechanical Shaker or Shaker Table

7.7 Vortex Mixer

7.8 15ml and 50ml Polyethylene Centrifuge tubes with caps

7.9 25ul, 50ul, 250ul, and 500ul syringes

7.10 10ml, 25ml, 250ml, and 1000ml Polyethylene graduated cylinder

7.11 Volumetric Pipettor and tips

7.12 1ml and 5ml Disposable polyethylene luer lock syringes

7.13 0.2um Nylon syringe filter 13mm and 25mm

7.14 10ml, 25ml, and 100ml Polyethylene or Polypropylene volumetric flasks

7.15 Disposable polyethylene transfer pipettes

7.16 2.0ml polyethylene screw cap vials

7.17 HDPE Wash Bottles

7.18 Spatula – Stainless Steel or Wood

7.19 Top loading balance – capable of weighing samples to +/- 0.01 grams

7.20 Centrifuge – 3000 rpm minimum speed

7.21 Micro-scoop 10mg

7.22 Nitrogen Evaporator, TurboVap LV or ExcelVap

8.0 STANDARDS AND REAGENTS

8.1 Methanol – HPLC grade or equivalent

8.2 Reagent water – HPLC grade or equivalent - free of interference

8.3 SPE Cartridges – Wax (weak anion exchange) or equivalent

8.4 Acetic Acid - HPLC grade or equivalent

8.5 Ammonium Hydroxide – Fisher A669-212 or equivalent (28-30% Aqueous Ammonia)

8.6 3% v:v Ammonium Hydroxide Solution – add 10ml of 30% Ammonium Hydroxide to 90ml of reagent water. Store at room temperature for up to 1 month.

8.7 0.3% v:v Ammonium Hydroxide in Methanol Solution – mix 1ml NH4OH and 99ml Methanol (based off 30%).

8.8 1% v:v Ammonium Hydroxide in Methanol Solution – mix 3.3ml NH4OH and 97ml Methanol (based off 30%).

8.9 Dilution Mix - Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid

8.10 Formic Acid – ACS grade or equivalent

8.10.1 0.1M Formic acid – add 4.6g formic acid to 1L of reagent water. Store at room temperature for up to a year.

8.10.2 0.3M Formic acid – add 13.8g formic acid to 1L of reagent water. Store at room temperature for up to a year.

8.10.3 5% v:v Formic acid – mix 5ml formic acid and 95ml of reagent water. Store at room temperature for up to a year.

8.10.4 50% v:v Formic acid – mix 50ml formic acid and 50ml of reagent water. Store at room temperature for up to a year.

8.10.5 1:1 Formic Acid in Methanol Solution – mix 50ml 0.1M formic acid and 50ml of methanol. Store at room temperature for up to a year.

8.11 ENVI-carb – graphitized carbon powder

8.12 PFAS EIS Mix – prepared in methanol at various concentrations by the vendor. All EIS solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

8.13 PFAS Spike Solution – prepared in methanol at various concentration by the LC/MS/MS analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

8.14 PFAS NIS Mix - prepared in methanol at various concentrations by the vendor. All EIS solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use

9.0 INTERFERENCES

9.1 Samples expected to contain high levels of PFAS compounds should be screened prior to extraction.

9.2 The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) or Teflon products, HPLC solvent lines, methanol, aluminum foil, SPE transfer lines, bottle caps, etc. All the materials used for this method must be demonstrated to be free from interferences.

9.3 Contact with glass containers, pipettes, or syringes should be minimized since the PFAS compounds can potentially adsorb (stick) to glass surfaces.

9.4 SPE cartridges can be a source of interferences. The analysis of method and field blanks can provide important information regarding the presence or absence of such interferences. Brand and lots of SPE devices should be tested to ensure that contamination does not preclude analyte identification and quantitation.

9.5 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the soil or sediment. Humic and/or fulvic material can be co-extracted and can cause enhancement and/or suppression in the electrospray ionization source.

10.0 PROCEDURE

10.1 The extraction of all samples must be documented on a “prep sheet”. The prep sheet will include such items as: batch number, sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, extraction dates and times, and extraction technician. The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the LC/MS/MS analyst with the extracts. The Batch number, extraction technician, and extraction start Date and Time are entered into LIMS.

10.2 Remove any foreign objects such as twigs or rocks. Thoroughly mix the sample with a spatula. Refer to SOP QA034 for more information on sample homogenization. Do not homogenize samples in glass containers or on aluminum tray.

10.3 Transfer approximately 5.0 gram of each soil or sediment sample to the appropriately labeled 50ml centrifuge tube. Use a clean spatula for each sample. Record the weight to the nearest 0.01 gram on the prep sheet.

> **Note**: If the samples are biosolids or similar matrix use 0.5 gram. These samples should be prepared in their own batch with separate 0.5-gram QC samples.

10.4 It may be beneficial to add a small amount (10% of sample weight or less) of PFAS free water to unusually dry samples.

10.5 Use 5.0 gram of blank sand wetted with 2.5 gram (2.5ml) of PFAS free water for the method blank (MB), blank spike (BS) low level blank spike (LLBS). Use additional 5.0 gram aliquots for the matrix spike (MS) and matrix spike duplicate (MSD).

> **Note**: If the samples are biosolids or similar matrix use 0.5 gram of blank sand wetted with 0.25 gram (0.25ml) of PFAS free water for the method blank (MB), blank spike (BS) low level blank spike (LLBS). Use additional 0.5-gram aliquots for the matrix spike (MS) and matrix spike duplicate (MSD).

10.6 Using the dedicated surrogate syringe add **25ul** of isotope dilution standard to each of the samples including the QC samples. Record the isotope dilution standard lot number on the prep sheet. Cap and vortex the samples to mix.

10.7 Using the dedicated spike syringe or volumetric pipettor add **32ul** of PFAS spike solution to the LLBS. Record the spike lot numbers on the prep sheet. Cap and vortex the samples to mix.

10.8 Using the dedicated spike syringe or volumetric pipettor add **200ul** of PFAS spike solution to the BS, MS, and MSD. Record the spike lot numbers on the prep sheet. Cap and vortex the samples to mix.

10.9 Allow the samples and QC so equilibrate for at least 30 minutes before extracting.

10.10 Add 10ml of 0.3% ammonium hydroxide in methanol solution to each centrifuge tube. Cap and vortex each sample to thoroughly mix the contents.

10.11 Place the centrifuge tubes on the shaker table and shake for 30 minutes.

10.12 Remove the samples from the shaker table. Centrifuge the samples for 10 minutes at 2800 rpm to separate the solids from the extract.

10.13 Transfer supernatant to a clean appropriately labelled 50ml Centrifuge tube.

10.14 Add an additional 15ml of 0.3% ammonium hydroxide in methanol solution to the sample, cap and vortex.

10.15 Place the centrifuge tubes on the shaker table and shake for 30 minutes.

10.16 Remove the samples from the shaker table. Centrifuge the samples for 10 minutes at 2800 rpm to separate the solids from the extract.

10.17 Transfer the supernatant from the second extraction into the centrifuge tube with the supernatant from the fist extraction.

10.18 Add an additional 5ml of 0.3% ammonium hydroxide in methanol solution to the sample, cap and vortex.

10.19 Centrifuge the samples for 10 minutes at 2800 rpm to separate the solids from the extract.

10.20 Combine the final supernatant in the 50ml Centrifuge tube with the supernatant from the first and second extraction.

10.21 Using a 10mg micro-scoop, add 10mg of ENVI-Carb powder to each sample and QC extract. Cap and vortex each sample to thoroughly mix the contents.

NOTE: Excessive contact time with the carbon (more than 5 minutes) may cause low recoveries.

10.22 Label another 50ml centrifuge tube for each sample and QC extract.

10.23 Centrifuge the extracts for 10 minutes at 2800 rpm to separate the ENVI-Carb from the extract. Immediately decant the extracts into the new centrifuge tube. Rinse the original tube with a small amount of 0.3% ammonium hydroxide in methanol being careful not to disturb the carbon at the bottom. Transfer the rinse to the new tube.

10.24 Place the centrifuge tubes in the rack for the nitrogen evaporator. The temperature of the evaporator should be set to ~55°C and the nitrogen flow rate to ~1.2 ml/min. Concentrate the extract to 7-8 ml.

NOTE: Evaporation of all the methanol can cause significant loss of the neutral compounds.

10.25 Label a 125ml HDPE bottle for each sample and QC extract. Fill each bottle with approximately 100ml of PFAS free water.

10.26 Transfer the extracts into the 125ml HDPE bottles. Rinse the original tube with a small amount of PFAS free water and transfer that to the bottle. This will result in an extract solution that is less than 10% methanol.

10.27 Check the pH of each extract by dipping a disposable polyethylene transfer pipette into the extract and touching it to the pH paper. Record the pH on the prep sheet.

10.28 The pH should be 6.5 +/- 0.5. If necessary, adjust the pH with 50% formic acid or 30% ammonium hydroxide solution OR with 5% formic acid or 3% ammonium hydroxide solution.

10.29 Assemble the solid-phase extraction system.

10.30 Label the side of each cartridge with the sample ID.

10.31 Loosely pack deactivated glass wool to half the height of the SPE cartridge and attach each of them to the SPE manifold.

10.32 Condition each SPE cartridge with 15ml of 1% ammonium hydroxide in methanol solution followed by 5ml of 0.3M formic acid. Use gravity flow if possible or a 1 to 2 ml/min flow rate. Stop the flow just before the cartridge goes dry.

Note: Do not allow the cartridge to go dry.

10.33 Using a reservoir adaptor, attach the sample reservoir to the top of the SPE cartridge.

10.34 Transfer an aliquot of each extract including the QC extracts to the appropriate sample reservoirs.

10.35 Turn on the vacuum and draw the extract through the cartridge at a rate of about 5 ml/min. Add additional extract aliquots to the sample reservoirs until the entire extract has passed through the cartridge. If particulates clog the cartridge, increase the vacuum to maintain a reasonable flow rate.

10.36 Once the entire extract has been pulled through the cartridge, shut off the vacuum. Rinse each sample bottle and reservoir with 2 x 5ml aliquots of reagent water. Draw the reagent water through the cartridge. Shut off the vacuum once the water has passed through the cartridge.

10.37 Rinse each sample bottle and reservoir with 5ml of the 1:1 0.1M Formic Acid/methanol solution. Turn on the vacuum. Draw the solution through the cartridge. Dry the cartridge by pulling air through the cartridge for another 15 seconds. Shut off the vacuum.

10.38 Open the SPE manifold and place an appropriately labeled 15.0ml centrifuge tube in

the rack under the position for SPE cartridge.

10.39 Set the manifold top back on the system, make sure that each of the delivery tubes goes into the appropriate centrifuge tube.

10.40 Rinse each sample bottle and reservoir with 5ml of 1% ammonium hydroxide in methanol solution. Use a pipet to transfer the solution to the SPE cartridge. Allow it to pass through the cartridge under gravity flow, then apply a slight vacuum to draw the remaining solution through the cartridge.

10.41 Open the SPE manifold and remove all the centrifuge tubes. If necessary, adjust the volume to 5ml with 1% ammonium hydroxide in methanol solution.

10.42 Add 25ul of NIS solution to each centrifuge tube. Then add 25ul of concentrated acetic acid to each centrifuge tube, cap and vortex.

10.43 Label another 15ml centrifuge tube for each sample and QC extract.

10.44 Attach 0.2um syringe filters to 5ml polypropylene syringes. Remove the barrel and pour the entire 5ml extract into the syringe. Insert the barrel and filter each extract into the appropriately labeled centrifuge tube.

10.45 Cap each centrifuge tube. Transfer the extracts to the LCMSMS lab for storage.

10.46 Store the extracts at ≤6°C. Extracts must be allowed to come to room temperature prior to analysis. All extracts should be vortexed just prior to transfer to the autosampler vials.

10.47 Determination of %solids is performed in accordance with the SOP EGN007 for Percent solids in solid matrix.

11.0 QC REQUIREMENTS

11.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

11.2 A method blank (MB), blank spike (BS), low-level blank spike (LLBS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples. If there is insufficient sample to extract a matrix spike duplicate (MSD) then a sample duplicate (DUP) should be extracted.

12.0 DOCUMENTATION REQUIREMENTS

12.1 See analytical SOP EMS1633

13.0 DATA REPORTING

13.1 See analytical SOP EMS1633

14.0 POLLUTION PREVENTATION & WASTE MANAGEMENT

14.1 Waste methanol is placed in the “non-chlorinated waste” container.

14.2 Spent solid-phase extraction cartridges may be disposed of in the trash.

14.3 Extracted soil samples are placed in a waste container after the solvent has drained.

14.4 Waste soil from the homogenizing process should be placed in the “soil waste” container. NOTE: Waste soil from foreign soils must follow “foreign soil” disposal requirements.

14.5 Samples are archived and stored for 30 days after analysis. After the storage time has elapsed, the remaining aqueous samples are transferred to the appropriate drums for disposal.

15.0 REFERENCES

15.1 EPA Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, January 2024

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
references	Revised to final 1633 method
10.24	Revised concentrate to 7-8 ml

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
00	01/05/2023	ANITA JAGTAP
01	07/19/2023	Olga Azarian
02	2/22/24	Raj Singh

END OF DOCUMENT

LAB SUPERVISOR: Raj

QA OFFICER: Jaffar

EFFECTIVE DATE: 03/21/2024

TITLE: STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF PER- AND POLYFLUORINATED ALKYL SUBSTANCES FROM WATER SAMPLES FOR LC/MS/MS ANALYSIS

REFERENCES: EPA METHOD 1633, January 2024

REVISED SECTIONS: references, 8.6, 8.7, 8.8, 8.9,15.1

1.0 SCOPE AND APPLICATION

1.1 This procedure applies to low-level aqueous samples submitted for Per- and Polyfluorinated Alkyl Substances (PFAS) analysis by LC/MS/MS using the Isotope Dilution technique.

2.0 SUMMARY OF METHOD

2.1 A 500ml aliquot of the sample (entire bottle) is extracted utilizing a solid phase extraction cartridge. The cartridge is eluted with basic methanol. The extract is carbon cleaned, filtered and the final volume is adjusted to 5.0ml, and then transferred to a centrifuge tube for storage.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 See analytical SOP-EMS1633

4.0 DEFINITIONS

4.1 Batch: A group of samples that are similar with respect to the matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours whichever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Low Level Blank Spike (LLBS): An analyte-free matrix spiked with a known amount of analyte(s) at 2x LLOQ, processed simultaneously with the samples through all the steps of the analytical procedure. Low-Level Blank Spike Recoveries are used to document laboratory performance at the LLOQ for a given method. This may also be called a

Low Level Laboratory Control Sample (LLLCS) or the Low-Level Ongoing Precision And Recovery Standard (LLOPR).

4.4 Extracted Internal Standards (EIS): A standard containing isotopically labelled versions of the native target analytes. These isotopes are usually labelled with C13, d2, or O18 atoms. Isotope Dilution Standards are used to measure the extraction efficiency and to correct the concentrations of the native analytes based on the recovery of their isotopically labelled analogs.

4.5 Field Blank (FB): An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are present in the field environment.

4.6 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and are still considered valid.

4.7 Matrix Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.9 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.10 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Laboratory Safety Manual which includes the use of Safety glasses, gloves and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the SDS prior to using any new reagents or solvents.

5.3 Methanol is an inhalation hazard. Use in a well-ventilated area.

5.4 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets is available to all personnel involved in these analyses.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIME

6.1 Preservation

6.1.1 Samples shall be collected in 500ml HDPE bottles fitted with a polyethylene screw cap. Alternate size HDPE bottles may be used depending on project requirements. **Glass bottles with Teflon lined caps can NOT be used.**

6.1.2 The samples must be chilled to ≤6°C from the time of collection until arrival at the laboratory. The samples must be refrigerated at ≤ 6°C or frozen at ≤ -20°C from the time of receipt until extraction.

6.1.3 The extracts should be stored at ≤6°C. They must be allowed to come to room temperature prior to analysis. All extracts should be vortexed just prior to transfer to the autosampler vials.

6.2 Holding Time

6.2.1 Aqueous samples must be extracted and analyzed within 28 days of collection if stored at ≤6°C or within 90 days of collection if stored at ≤ -20°C. The Date/Time that the extraction is started and completed must be recorded on the prep sheet.

7.0 APPARATUS AND MATERIALS

7.1 Solid-phase cartridge extraction system – suitable for use with extraction cartridges

7.2 Vacuum pump

7.3 Vacuum Flasks – or equivalent

7.4 SPE reservoirs – various sizes

7.5 SPE cartridges – Weak Anion Exchange – must have pKa of >8 and 150mg bed size

7.6 15ml and 50ml Polyethylene Centrifuge tubes with caps

7.7 10ml, 25ml, 250ml, and 1000ml Polyethylene graduated cylinder

7.8 Top loading balance for aqueous sample volume determination to 0.1 g

7.9 10ul, 25ul, 50ul, 250ul and 500ul syringes

7.10 Volumetric Pipettors and tips

7.11 1ml and 5ml Disposable polyethylene luer lock syringes

7.12 0.2um Nylon syringe filter 13mm and 25mm

7.13 10ml, 25ml, and 100ml Polyethylene or Polypropylene volumetric flasks

7.14 Disposable polyethylene transfer pipettes

7.15 Deactivated glass wool.

7.16 2.0ml polyethylene screw cap vials

7.17 HDPE Wash Bottles

7.18 Nitrogen Evaporator, TurboVap LV or ExcelVap

7.19 Balance +/- 0.1 gram

7.20 Centrifuge - 3000 rpm minimum speed

7.21 Micro-scoop 10mg

8.0 STANDARDS AND REAGENTS

8.1 Methanol – HPLC grade or equivalent

8.2 Reagent water – HPLC grade or equivalent - free of interference

8.3 SPE Cartridges – Wax (weak anion exchange) or equivalent

8.4 Acetic Acid - HPLC grade or equivalent

8.5 Ammonium Hydroxide – Fisher A669-212 or equivalent (28-30% Aqueous Ammonia)

8.6 3% v:v Ammonium Hydroxide Solution – add 10ml of 30% Ammonium Hydroxide to 90ml of reagent water. Store at room temperature and replace after 3 months.

8.7 0.3% v:v Ammonium Hydroxide in Methanol Solution – mix 1ml NH4OH and 99ml Methanol (based off 30%). Store at room temperature and replace after 1 month.

8.8 1% v:v Ammonium Hydroxide in Methanol Solution – mix 3.3ml NH4OH and 97ml Methanol (based off 30%). Store at room temperature and replace after 1 month.

8.9 Dilution Mix - Methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid-– add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL). Store at room temperature and replace after 1 month.

8.10 Formic Acid – ACS grade or equivalent

8.10.1 1M Formic acid – add 4.6g formic acid to 1L of reagent water. Store at room temperature for up to a year.

8.10.2 0.3M Formic acid – add 13.8g formic acid to 1L of reagent water. Store at room temperature for up to a year.

8.10.3 5% v:v Formic acid – mix 5ml formic acid and 95ml of reagent water. Store at room temperature for up to a year.

8.10.4 50% v:v Formic acid – mix 50ml formic acid and 50ml of reagent water. Store at room temperature for up to a year.

8.10.5 1:1 Formic Acid in Methanol Solution – mix 50ml 0.1M formic acid and 50ml of methanol. Store at room temperature for up to a year.

8.11 ENVI-carb – graphitized carbon powder

8.12 PFAS EIS Mix – prepared in methanol at various concentrations by the vendor. All EIS solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

8.13 PFAS Spike Solution – prepared in methanol at various concentration by the LC/MS/MS analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

8.14 PFAS NIS Mix - prepared in methanol at various concentrations by the vendor. All EIS solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

9.0 INTERFERENCES

9.1 Samples expected to contain high levels of PFAS compounds should be screened prior to extraction.

9.2 The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) or Teflon products, HPLC solvent lines, methanol, aluminum foil, SPE transfer lines, bottle caps, etc. All materials used for this method must be demonstrated to be free from interferences.

9.3 Contact with glass containers, pipettes, or syringes should be minimized since the PFAS compounds can potentially adsorb (stick) to glass surfaces.

9.4 SPE cartridges can be a source of interferences. The analysis of method and field blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices should be tested to ensure that contamination does not preclude analyte identification and quantitation.

9.5 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending

upon the nature of the water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent.

10.0 PROCEDURE

10.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: batch number, sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, extraction dates and times, and extraction technician. The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the LC/MS/MS analyst with the extracts. The Batch number, extraction technician, and extraction start Date and Time are entered into LIMS.

10.2 The 150mg WAX (weak anion exchange) SPE cartridge is considered the default cartridge for this method. Other bed sizes may be used if they have been fully validated.

10.2.1 Assemble the solid-phase extraction system.

10.2.2 Label the side of each cartridge with the sample ID.

10.2.3 Loosely pack deactivated glass wool to half the height of the SPE cartridge and attach each of them to the SPE manifold.

10.2.4 Condition each SPE cartridge with 15ml of 1% ammonium hydroxide in methanol solution followed by 5ml of 0.3M formic acid. Use gravity flow if possible or a 1 to 2 ml/min flow rate. Stop the flow just before the cartridge goes dry.

Note: Do not allow the cartridge to go dry.

10.2.5 Using a reservoir adaptor, attach the sample reservoir to the top of The SPE cartridge.

10.2.6 The volume of sample is determined by weighing the sample, bottle, and cap and recording the weight to 0.1g. Air dry the empty bottle after the bottle is rinsed, the rinse is transferred. Weigh the empty bottle with the cap on and subtract from the weight with the sample.

10.2.7 Use 500ml HDPE bottles for the method blank (MB), blank spike (BS) and Low- Level Blank Spike (LLBS). Fill each of these bottles with 500ml of reagent water. Use 100ml HDPE bottles for MB, BS, LLBS if the project required smaller sample volumes.

10.2.8 Use separate bottles for the matrix spike (MS) and the matrix spike duplicate (MSD). **NOTE: Bottles must NOT be split.** If there are no samples with two extra bottles for the MS/MSD, then prepare a matrix spike (MS) and a duplicate (DUP) from separate samples. Record the sample ID, bottle number, and volume on the prep sheet.

10.2.9 Using the dedicated surrogate syringe add **25ul** of isotope dilution standard to each of the samples including the QC samples. Record the isotope dilution standard lot number on the prep sheet. Cap and invert the samples to mix.

10.2.10 Using the dedicated spike syringe or volumetric pipettor add **32ul** of PFAS spike solution to the LLBS. Record the spike lot numbers on the prep sheet. Cap and invert the samples to mix.

10.2.11 Using the dedicated spike syringe or volumetric pipettor add **200ul** of PFAS spike solution to the BS, MS, and MSD. Record the spike lot numbers on the prep sheet. Cap and invert the samples to mix.

10.2.12 Check the pH of each sample by dipping a disposable polyethylene transfer pipette into the sample and touching it to the pH paper. Record the pH on the prep sheet.

10.2.13 The pH should be 6.5 +/- 0.5. If necessary, adjust the pH with 50% formic acid or 30% ammonium hydroxide solution OR with 5% formic acid or 3% ammonium hydroxide solution. Record this on the prep sheet.

10.2.14 Transfer an aliquot of each sample including the QC samples to the appropriate sample reservoirs.

10.2.15 Turn on the vacuum and draw the sample through the cartridge at a rate of about 5 ml/min. Add additional sample aliquots to the sample reservoirs until the entire sample has passed through the cartridge. As particulate clogs the cartridge, increase the vacuum to maintain a reasonable flow rate.

10.2.16 Once the entire sample has been pulled through the cartridge, shut off the vacuum. Rinse each sample bottle and reservoir with 2 x 5ml aliquots of reagent water. Draw the reagent water through the cartridge. Shut off the vacuum once the water has passed through the cartridge.

10.2.17 Rinse each sample bottle and reservoir with 5ml of the 1:1 0.1M Formic Acid/methanol solution. Turn on the vacuum. Draw the solution through the cartridge. Dry the cartridge by pulling air through the cartridge for another 15 seconds. Shut off the vacuum.

10.2.18 Open the SPE manifold and place an appropriately labeled 15.0ml centrifuge tube in the rack under the position for SPE cartridge.

10.2.19 Set the manifold top back on the system, make sure that each of the delivery tubes goes into the appropriate centrifuge tube.

10.2.20 Rinse each sample bottle and reservoir with 5ml of 1% ammonium hydroxide in methanol solution. Use a pipet to transfer the solution to the SPE cartridge. Allow it to pass through the cartridge under gravity flow, then apply a slight vacuum to draw the remaining solution through the cartridge.

10.2.21 Retain the sample bottle and cap for Initial Volume determination. See Section 10.2.6.

10.2.22 Open the SPE manifold and remove all the centrifuge tubes. If necessary, adjust the volume to 5ml with 1% ammonium hydroxide in methanol solution. Add 25ul of concentrated acetic acid to each centrifuge tube, cap and vortex to mix.

10.2.23 Using a 10mg micro-scoop, add 10mg of ENVI-Carb powder to each sample and QC extract. Cap and vortex each sample to thoroughly mix the contents.

Note: Excessive contact time with the carbon (more than 5 minutes) may cause low recoveries.

10.2.24 Centrifuge the samples for 10 minutes at 2800 rpm to separate the ENVI-Carb from the extract.

10.2.25 Label another 15ml centrifuge tube for each sample and QC extract. Add 25ul of NIS solution to each centrifuge tube.

10.2.26 Attach 0.2um syringe filters to 5ml polypropylene syringes. Remove the barrel and pour the entire 5ml extract into the syringe. Insert the barrel and filter each extract into the appropriately labeled centrifuge tube.

10.2.27 Cap each centrifuge tube. Transfer the extracts to the LCMSMS lab for storage.

10.2.28 Store the extracts at ≤4°C. Extracts must be allowed to come to room temperature prior to analysis. All extracts should be vortexed just prior to transfer to the autosampler vials.

11.0 QC REQUIREMENTS

11.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

11.2 A method blank (MB), blank spike (BS), low-level blank spike (LLBS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples. If there is insufficient sample to extract a matrix spike duplicate (MSD) then a sample duplicate (DUP) should be extracted.

12.0 DOCUMENTATION REQUIREMENTS

12.1 See analytical SOP EMS1633

13.0 DATA REPORTING

13.1 See analytical SOP EMS1633

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Waste methanol is placed in the “non-chlorinated waste” container.

14.2 Spent solid-phase extraction cartridges may be disposed of in the trash.

14.3 Extracted soil samples are placed in a waste container after the solvent has drained.

14.4 Waste soil from the homogenizing process should be placed in the “soil waste” container. NOTE: Waste soil from foreign soils must follow “foreign soil” disposal requirements.

14.5 Samples are archived and stored for 30 days after analysis. After the storage time has elapsed, the remaining aqueous samples are transferred to the appropriate drums for disposal.

15.0 REFERENCES

15.1 EPA Method 1633, Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, January 2024.

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
8.6,8.7,8.8	Added expiration date
8.9	Added preparation and expiration date
15.1	Updated the method reference to the most current version

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	REVISED BY
00	01/05/2023	ANITA JAGTAP
01	7/19/2023	Olga Azarian
02	02/22/2024	Raj Singh

END OF DOCUMENT

LAB SUPERVISOR: _____

QA OFFICER: _____

EFFECTIVE DATE: 9-27-2021

TITLE: PREPARATION OF SEMI-VOLATILE EXTRACTABLES IN AQUEOUS SAMPLES (BASE, NEUTRAL, ACID ORGANICS; PESTICIDES, POLYCHLORINATED BIPHENYLS, DIESEL RANGE ORGANICS, EXTRACTABLE PETROLEUM HYDROCARBONS)

REFERENCES: SW846 3510C (Revision 3, December 1996)
ADDED SECTIONS: 10.3.9.1.1.1

1.0 SCOPE AND APPLICATION

1.1 This method describes the extraction procedure of semi-volatile organic compounds in various water sources for analysis by gas chromatography (GC) or gas chromatography/mass spectrometry (GC/MS).

2.0 SUMMARY

2.1 Extract a measured volume of sample, usually 1 liter, with an immiscible organic solvent (methylene chloride or hexane) using a separatory funnel. Serial extractions are performed at specified pHs. The extract is concentrated by evaporation/distillation by a factor of 100 – 1,000 prior to analysis to increase the apparent concentration of target analytes and improve the sensitivity of the instrumental technique. This method is applicable for EPA methods 608, 625, and SW846 8081, 8082, 8270, 8015C, EPH, and NJ OQA 25.

2.2 An option for preparing aqueous samples using a reduced initial volume (~250ml) for analysis by a large volume injection (LVI) technique is also described.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 See determinative method.

4.0 DEFINITIONS

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. Types of Blanks may include: Method Blank, Instrument Blank, Storage Blank, and Leachate Blank.

BLANK SPIKE (BS) - An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

BATCH - A group of samples and associated quality control samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

CLASS A GLASSWARE - Volumetric laboratory glass that has been manufactured, calibrated and certified to established ASTM volume standards. Under normal laboratory conditions, Class A Glassware does not require volume calibration or verification.

EXTRACTION – The process of removing a desired compound from a matrix using chemical or mechanical procedures. The process is used to isolate and concentrate targeted constituents for instrumental analysis

HOLDING TIME - The maximum time that samples may be held prior to preparation and/or analysis and still are considered valid.

KUDERNA DANISH (K-D) – A three-stage glass solvent concentration device consisting of a large volume receiving flask (250 or 500mL), a small volume receiver and a three-ball air cooled condenser (Snyder column). This device is used to evaporate large volumes of solvent used for organic extractions to increase the concentration of the analyte in the solvent.

MATRIX - The predominant material of which a sample is composed. For the purpose of this method, a sample matrix is either water or soil/sediment. Matrix is <u>not</u> synonymous with phase (liquid or solid).

MATRIX SPIKE (MS) - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE (MSD) - a second aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

METHOD BLANK (MB) - an analytical control consisting of all reagents, internal standards, and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

REAGENT WATER – Purified water in which an interferant is not observed at or above the minimum reporting limit of the parameters of interest.

SEPARATORY FUNNEL – A large volume (500, 1000 or 2000mL) closed funnel used for separating immiscible liquids from each other. The device is used for shaking samples with solvent to extract organic constituents. One of the phases of the mixture is removed through the stopcock opening in the base to effect the separation.

SURROGATE - An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

WATER BATH – A waterbath equipped with an evaporation collection system. The system collects the extraction solvent which is then disposed via the appropriate waste stream.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Laboratory Safety Manual and SGS Safety Policy, which includes the use of safety glasses and lab coats. Handle all acids, which are corrosive with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, treat each chemical as a potential health hazard. Reduce exposure to these reagents to the lowest possible level. The laboratory is responsible for maintaining a current file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets is available to all personnel involved in these analyses.

6.0 INTERFERENCES

6.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. Demonstrate that these materials are free from interferences under the conditions of the analysis by analyzing method blanks.

6.2 Interferences co-extracted from the samples will vary considerably from source to source. If interferences prevent the analysis of an extracted sample, further cleanup of the sample extract may be necessary.

6.3 Phthalate esters contaminate many products commonly used in the laboratory. Avoid plastics in particular because they contain phthalates, used as plasticizers, which can leach from these materials. Practice sound, consistent materials control to avoid phthalate contamination, which may occur at any time.

6.4 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Specifically, Aldrin, Heptachlor, and most organophosphorus pesticides will degrade in this situation. This occurs in glassware that is difficult to rinse. Carefully hand-rinse these items to avoid this problem.

7.0 COLLECTION, PRESERVATION AND HOLDING TIMES

7.1 Collect EPA 608, 625, SW-846 8081, 8082, 8270 samples in 1-liter amber glass bottles without preservatives. Collect SW-846 8015 and BNJEPH samples in 1-liter amber glass bottles preserved with hydrochloric acid. Samples for NJ OQA25 are preserved with HCl upon collection.

7.2 1 liter (nominal) or 250ml-LVI of unpreserved sample is required for extraction with the exception of NJ OQA 25 samples. Additional sample volume is necessary for any samples used for matrix duplicates and matrix spikes. Therefore, submit 3 liters of at least one sample in every group of 20 field samples for analysis to accommodate all quality control requirements.

7.3 The samples must be protected from light and refrigerated at 0 to <6° C from the time of receipt until extraction and analysis.

7.4 For methods EPA 625; SW-846 8081, 8082, 8270 & 8015C. Extract aqueous samples within 7 days of sampling and analyze within 40 days of the extraction.

7.5 For method 608, check the sample pH and adjust to 5-9 if necessary. Extract samples within 7 days of sampling and analyze within 40 days of the extraction. If the sample pH cannot be adjusted between 5 - 9, the sample must be extracted within 72 hours.

7.6 For Methods 608 and 625, check the sample for residual chlorine using test paper. If the sample tests positive add 80mg of sodium thiosulfate per liter of sample.

8.0 APPARATUS & MATERIALS

8.1 Amber Glass Bottle- 1000mL, 250mL

8.2 Separatory funnels - 2 liter with Teflon stopcocks.

8.3 Class A Volumetric Flasks: 1mL, 5mL, 10mL, 100mL volumes

8.4 One (1) ml volumetric pipettes or pre-calibrated 1ml syringes

8.5 Graduated cylinder- 10mL & 1000mL, Class A

8.6 Beaker- 250mL

8.7 4 position Automatic Separatory Funnel Shaker

8.8 Drying column – used for removing aqueous fraction from organic fraction. Composed of funnel (large or small) containing glass wool or filter paper and sodium sulfate

8.9 Waterbath with solvent evaporation collection system- large volume concentrator capable of capturing evaporated solvent. Composed of waterbath, chiller, and solvent collector

8.10 Porous Boiling Chips or Glass Beads

8.11 Kuderna-Danish sample collection setup- for waterbath with solvent evaporation collection system. Composed of Kuderna-Danish flask (500mL or 250mL), receiver (10mL, Class A calibration), and retaining clip

8.12 Snyder Column- three ball

8.13 Buchi Concentrator System- solvent concentrator capable of capturing evaporated solvent. Composed of Syncore Unit, Vacuum Pump, Chiller, & Recirculating Pump

8.14 Buchi tube, Class A- for Buchi concentrator system.

8.15 N-VAP Concentrator- small volume concentrator. Composed of waterbath and Nitrogen gas blow down apparatus.

8.16 Microsnyder Concentrator- small volume concentrator. Composed of waterbath and microsnyder column.

8.17 Vortex Mixer

8.18 2mL graduated amber glass vial and associated PTFE lined cap (screw top or crimp)

8.19 Refrigerator & Freezer- for sample and backup storage

8.20

9.0 REAGENTS

9.1 Reagent water - deionized and carbon filtered water prepared to ASTM Type II specifications.

9.2 Solvents - reagent grade for trace organic analysis. Each solvent lot must be checked for interferences prior to use. Refer to SOP EOP013 for the procedure regarding solvent lot approval.

9.2.1 Methylene chloride

9.2.2 Hexane

9.2.3 Acetone

9.2.4 Hydrochloric Acid

9.3 Solutions- generated from reagent grade for trace organic analysis solvents listed above or shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

9.3.1 Sodium hydroxide solution (10N) - purchase as a commercially prepared solution. Alternatively, dissolve 40g of NaOH in reagent water and dilute to 100 ml.

9.3.2 Sulfuric acid (1:1)- purchase as a commercially prepared solution (50% v/v). Alternatively prepare a 50% solution from equal parts concentrated sulfuric acid and reagent water.

9.4 Sodium sulfate - granular anhydrous, refer to EOP029 for preparation instructions.

9.5 Nitrogen Gas – High Purity Grade.

9.6 Surrogate and Matrix Spiking Solutions. See the analytical SOP for target compound specifications. See the extraction SOP for target compound amounts. Refer to the most current version of Form: OP026 for details on Standard and Spike preparation.

Note: Unopened stock solutions must be stored according to the manufacturers documented holding time and storage temperature recommendations. After opening, stock standards must be replaced after 6 months or sooner if manufacturer's expiration date comes first or comparison with quality control check samples indicate degradation.

9.7 pH indicator paper – wide range, minimum of pH 2 – pH 11.

9.8 Wide range residual and total chlorine test paper.

10.0 PROCEDURE

10.1 Assemble all needed apparatuses and rinse prior to use.

10.1.1 Separatory Funnel Preparation

10.1.1.1 Prepare previously washed 2L (or 500 ml for 3510C LVI) separatory funnel by closing valve and adding 5-10mL of DCM. Cap and shake until all inner surfaces have come in contact with the DCM. Discard methylene chloride by opening valve and decanting into chlorinated solvent waste container.

10.1.1.1.1 If water is present in separatory funnel, rinse with 5-10mL of acetone prior to methylene chloride prep.

10.1.1.1.2 Hexane rinse, if needed, may also be performed after the methylene chloride rinse.

10.1.2 Drying Column Preparation

10.1.2.1 Prepare a drying column by adding approximately 30-35g of baked sodium sulfate into a funnel lined with glass wool or filter paper. Rinse with 5-10 mL of methylene chloride, collecting excess into a waste container, and discard in chlorinated solvent waste.

10.1.3 Kuderna-Danish Sample Collection Setup Preparation

10.1.3.1 Rinse assembled Kuderna-Danish flask and receiver with 5-10mL of methylene chloride. Discard methylene chloride into chlorinated solvent waste.

10.1.3.2 Place a drying column on top of K-D flask to complete Kuderna-Danish Sample Collection Setup.

10.1.4 Snyder Column Preparation

10.1.4.1 Pass approximately 3-5mL of methylene chloride from top to bottom of Snyder Column and on the joints to rinse and prime. Collect excess methylene chloride and discard into chlorinated solvent waste.

10.1.5 Buchi Tube Sample Collection Setup Preparation

10.1.5.1 Prepare previously washed and dry Buchi Tubes by adding 5-10mL of DCM and shaking/swirling until all inner surfaces have come in contact with the DCM. Discard methylene chloride by pouring off into chlorinated solvent waste container.

10.1.5.1.1 If water is present, rinse with 5-10mL of acetone prior to methylene chloride prep.

10.1.5.1.2 Hexane rinse, if needed, may also be performed after the methylene chloride rinse.

10.1.5.2 Place a drying column on top of Buchi tube to complete Buchi Tube Sample Collection Setup

10.2 Turn on all equipment, check for proper operating conditions, and allow to reach operating temperatures prior to use.

10.2.1 Waterbath with Evaporation Collection System. System is composed of 2 parts: Waterbath and Condensation System (Chiller and Condensers). Each part will need to be checked prior to use.

10.2.1.1 Chiller

10.2.1.1.1 Check coolant level in chiller and top-up as needed.

10.2.1.1.2 Turn on chiller. Temperature of coolant should show up on digital display, and flowmeter will register rate of coolant flow in gallons per minute (gpm).

10.2.1.1.3 Check coolant lines for leaks. If any are noted, turn off chiller and notify team lead, supervisor, or manager.

10.2.1.1.4 Adjust flowrate of chiller to exceed flow rate listed on chiller operating parameter card.

10.2.1.1.5 Monitor coolant temperature and ensure it falls below the maximum chiller temperature listed on chiller operating parameter card. If it will not fall below maximum temperature, notify team lead, supervisor, or manager.

10.2.1.1.6 Continue to monitor coolant temperature throughout use. At any point of operation, if chiller temperature exceeds listed maximum, remove all samples and suspend use of the associated waterbath(s) until temperature falls below maximum. If it doesn't fall below maximum temperature, notify team lead, supervisor, or manager.

10.2.1.2 Waterbath

10.2.1.2.1 Check water level in waterbath. If water level is below ¾" of sample holding deck, top up with water.

10.2.1.2.2 Turn on waterbath. If waterbath will not turn on, notify team lead, supervisor, or manager.

10.2.1.2.3 Temperature of water in waterbath should show up on digital display. Check and adjust to ensure temperature is set to 75.0±5.0°C. Water set point temperature is never allowed to exceed 75.0°C. Water temperature is never allowed to exceed 80.0°C. Calibration of waterbath temperature occurs on a quarterly basis. Check calibration date- if expired, do not use. If waterbath will not turn on or out of calibration, notify team lead, supervisor, or manager.

10.2.1.2.4 Continue to monitor water temperature throughout use to ensure heating elements are operating normally and maintain water set point temperature. If waterbath does not maintain the water set point temperature, notify team lead, supervisor, or manager.

10.2.2 Buchi Concentrator System. System is composed of 2 parts: Buchi and Condensation System (Chiller and Condenser). Each part will need to be checked prior to use.

10.2.2.1 Refer to OP030 for proper Buchi Concentrator System Start-up instructions.

10.2.2.2 If any issues, notify a team lead, supervisor, or manager.

10.2.3 Microsnyder. System is composed of waterbath and microsnyder column. Waterbath will need to be checked prior to use.

10.2.3.1 Check water level in waterbath. If water level is below ¾" of sample holding deck, top up with water.

10.2.3.2 Turn on waterbath. If waterbath will not turn on, notify team lead, supervisor, or manager.

10.2.3.3 Temperature of water in waterbath should show up on digital display. Check and adjust to ensure temperature is set to 85.0±5.0°C. Microsnyder waterbath set point is higher due to lower volume of water. If full size waterbath is used, set point temperature is never allowed to exceed 75.0°C and water temperature is never allowed to exceed 80.0°C. If waterbath will not turn on, notify team lead, supervisor, or manager.

10.2.3.4 Continue to monitor water temperature throughout use to ensure heating elements are operating normally and maintain water set point temperature. If waterbath does not maintain the water set point temperature, notify team lead, supervisor, or manager.

10.2.4 N-VAP Concentrator. System is composed of 2 parts: Waterbath and Nitrogen Flow System. Each part will need to be checked prior to use.

10.2.4.1 Waterbath

10.2.4.1.1 Check water level in waterbath. If water level is below ¾" of top, top up with water.

10.2.4.1.2 Turn on waterbath. If waterbath will not turn on, notify team lead, supervisor, or manager.

10.2.4.1.3 Digital Display Waterbath. Temperature of water in waterbath should show up on digital display. Check and adjust to ensure temperature is set to 35.0±2.0°C. Water set point temperature is never allowed to exceed 37.0°C. If waterbath will not turn on, notify team lead, supervisor, or manager.

10.2.4.1.4 Manual Knob Waterbath. Temperature of water in waterbath will need to be manually calibrated and proper setting noted. Check and adjust to ensure temperature is set to 35.0±2.0°C. Water temperature is never allowed to exceed 37.0°C. If waterbath will not turn on or maintain proper temperature, notify team lead, supervisor, or manager.

10.2.4.1.5 Continue to monitor water temperature throughout use to ensure heating elements are operating normally and maintain water set point temperature. If waterbath does not maintain the water set point temperature, notify team lead, supervisor, or manager.

10.2.4.2 Nitrogen Flow System

10.2.4.2.1 Turn on flow of Nitrogen to system. Check to assure the incoming pressure does not exceed 30 pounds per square inch (psi) on pressure gauge.

10.2.4.2.2 Adjust flow rate to 10 liters per minute (lpm) on flow rotometer.

10.2.4.2.3 Check to ensure all needles are in good working order and clean.

10.2.4.2.4 If any issues, notify team lead, supervisor, or manager.

10.3 Pesticides/PCBs by EPA Method 608, SW846 Method 8081, SW846 Method 8082 (Sep Funnel, Water Bath, Buchi Concentrator, & N-VAP).

10.3.1 Confirm all samples present and delivered, listed on the LIMS batch sheet, and listed on preparation log. If any discrepancies, check with schedulers, team leads, supervisors, or managers.

10.3.2 If no discrepancies, document custody of samples in Internal Chain program and change status of batch to “ASN” reflecting the initiation of extraction in LIMS.

10.3.3 Label all apparatuses with the correct sample identification. Sample labels may transfer from one apparatus to the next.

10.3.4 Measure two 1- liter aliquots of deionized water for a method blank and blank spike into separate 1L amber glass bottles. For samples testing for both Pesticide and PCB (known as “shared batches”), two separate sets of spikes must be prepared. Therefore, measure four 1- liter aliquots of deionized water for 2 method blanks and 2 blank spikes into separate 1L amber glass bottles. Initial extraction pH must be 5 – 9. If pH is outside of the range adjust using sulfuric acid or sodium hydroxide.

10.3.5 Mark level of sample on outside of glass bottle. For samples filled to completion, a mark is placed on the cap.

10.3.6 Mix the sample thoroughly by shaking.

Note: Samples which do not homogenize, are multiphasic, have a gasoline or oil odor, thick aqueous sludges, visible oil layer, or gels may not be appropriate for this extraction.

These may require a reduced sample amount, alternate extraction technique, and comments to be added. Check with the team leads, supervisors, or managers for further instructions.

10.3.7 After mixing the sample, decant ¾" to 1" of sample into respective 2L separatory funnel. This is to allow for the addition of surrogate and/or spike.

10.3.8 Record the bottle number, sample description, and pH and chlorine (if needed) in the logbook.

10.3.9 Spiking & Surrogating. For proper execution protocols refer to EOP027.

10.3.9.1 Spiking Amounts. The test being requested will dictate the spike that will need to be added. Using a calibrated 1mL syringe, add spike to blank spike, matrix spike, and matrix spike duplicate (if present).

10.3.9.1.1 For Pesticide add 1mL of pesticide target compound spike.
10.3.9.1.1.1 For Pesticides by EPA 608.3, prepare additional spike with Toxaphene and Chlordane with every batch of 20 samples or less by adding 0.5 ml of the spike.

10.3.9.1.2 For Pesticide Low Level ("LL") add 0.1mL of pesticide target compound spike.

10.3.9.1.3 For PCB add 1mL of PCB target compound spike.

10.3.9.1.4 For PCB Low Level ("LL") add 1mL of PCB target compound spike.

10.3.9.1.5 For samples testing for both Pesticide and PCB (known as "shared batches"), two separate sets of blanks and spikes must be prepared. One for Pesticides and one for PCBs.

Note: Check the expiration date for all spiking materials to verify that the solution is valid. Do not use expired solutions for spiking.

Note: Other special spikes may be needed. Check the special spike section of the logbook for additional information.

10.3.9.2 Surrogate Amounts. The test being requested will dictate the amount of surrogate that will need to be added.

10.3.9.2.1 For Pesticide or PCB regular samples (not "LL"), add 1mL of the pesticide/PCB surrogate (labeled as "TCMX" on surrogate bottle) using a calibrated 1mL syringe to all samples, blanks, and spikes.

10.3.9.2.2 For Pesticide or PCB Low Level ("LL") samples, add 0.1mL of the pesticide/PCB surrogate (labeled as "TCMX" on surrogate bottle) using a calibrated 1mL syringe to all samples, blanks, and spikes

Note: Check the expiration date for all surrogating materials to verify that the solution is valid. Do not use expired solutions for surrogating.

Note: Other special surrogates maybe needed. Check the logbook for additional information.

10.3.10 Pour samples into their respective 2L separatory funnel.

10.3.11 Rinse the sample bottle with 60mL of methylene chloride. Transfer the 60mL methylene chloride rinse from the sample container into the separatory funnel containing the sample.

10.3.12 To determine the initial sample volume in the sample bottle, fill the empty sample jar with tap water to the mark on the jar. Pour the contents of the jar into a graduated cylinder and measure the volume. This is the sample volume that is recorded on the extraction log.

10.3.13 Hand shake the funnel vigorously for 15-20 seconds. Release excess pressure by venting the funnel into the hood by opening and closing the Teflon stopcock.

10.3.14 Extract the sample by shaking the funnel vigorously for 2 minutes either using the automatic shaker or by hand. Allow the aqueous solvent layers to separate for a minimum of 10 minutes.

10.3.15 Collect the methylene chloride layer (the bottom layer) by passing through the drying column into the assembled Kuderna-Danish.

Note: Additional sodium sulfate may be added as needed to prevent trace amounts of aqueous fraction present in methylene chloride layer from passing through.

Important: Do not allow the sodium sulfate to become dry between solvent extracts.

10.3.16 Rinse drying column with methylene chloride. This ensures quantative recovery of analytes of interest.

10.3.17 Add a second 60mL volume of methylene chloride to the separatory funnel. Seal and shake the separatory funnel and repeat the extraction/collection procedure described in Section 10.3.13 to 10.3.15. Perform a third extraction/collection in the same manner.

10.3.18 If needed, once all three 60mL volumes of methylene chloride have been added, centrifuge the solvent layer to break emulsions.

10.3.19 Once the three extraction steps are completed, perform a final rinse of the drying column with an additional 10mL of methylene Chloride. Allow the methylene chloride to drain through until dripping stops.

10.3.20 Remove drying column and discard sodium sulfate, glass wool or filter paper into solid sulfate waste.

10.3.21 Add in boiling chip and Snyder column. The sample is now ready to be concentrated.

10.3.22 Sample Concentration Procedure. Document the procedure in the extraction logbook.

10.3.22.1 Water Bath with Evaporation Collection System

Note: Fill in waterbath information, chiller information, and flow rates once samples have been loaded.

Note: If a sample ruptures into the waterbath, immediately close the hood, allow the boiling action of the water to subside, fumes to dissipate, and notify a team lead, supervisor, or manager. Do not attempt glassware removal or cleanup by yourself.

10.3.22.1.1 Position K-D flask in the waterbath. Immersing the 10mL concentrator tube into the waterbath. Concentrate samples to about 5-7mL.

10.3.22.1.2 Allow the K-D flask, receiver, and Snyder column to cool. During this process, approximately 1mL of methylene chloride will re-condense and flow down the side of the K-D flask removing most of the extract remaining in the flask and joints.

10.3.22.1.3 Remove Snyder column and slowly add 0.5-1mL of methylene chloride to the tilted K-D flask while rotating the flask. This is a final rinse of the flask and joint. Tilt upright to allow the methylene chloride to drain into the receiver.

10.3.23 Solvent Exchange. A solvent exchange to hexane is required for Pest/PCB samples.

10.3.23.1 Add 50mL of hexane into K-D setup and swirl to contact all surfaces.

10.3.23.2 Transfer sample by pouring from K-D setup into Buchi tube.

10.3.23.3 Buchi Concentrator System

Note: In logbook, fill in Buchi concentrator information, chiller information, and flow rates once samples have been loaded

10.3.23.3.1 Load Buchi tubes into Buchi Concentrator. Use yellow plugs to close any Buchi tube slots that are not being used.

10.3.23.3.2 Select program “Pest/PCB Hexane X”, and press “Start” on the vacuum controller. Turn rotation knob until digital display reads 250 rotations per minute. The extract is concentrated to approximately 1mL.

10.3.23.3.3 Once program is finished, press “Stop” on vacuum controller and turn rotation knob to 0 rotations per minute.

10.3.23.3.4 Unload Buchi tubes from Buchi Concentrator and allow to cool.

10.3.24 Final volume adjustment. Samples may require further concentration to reach proper volumes.

10.3.24.1 For Methods 608, final volume is 5mL. For Methods 8081 and 8082, final volume is 10mL. For 608 Low Level (“608 LL”), 8081 Low Level (“8081 LL”),

and 8082 Low Level ("8082 LL), final volume is 1mL. If samples will not concentrate to final volumes or if they change states (i.e. liquid to gel), notify team lead, supervisors, and managers for further instructions. These may require a modified sample amount, alternate concentration technique, and comments to be added.

10.3.24.2 N-VAP Method. Immerse the nipple of the Buchi tube in the waterbath of the N-VAP. Direct a gentle stream of nitrogen gas onto the extract by adjusting height of needle until small dimple appears on surface. Evaporate the solvent until the desired volume is reached. Occasionally rinse the sidewalls of the concentrator tubes with methylene chloride during the evaporation step.

10.3.25 Vialing

10.3.25.1 Sample Vial Labeling

10.3.25.1.1 For 608 Low Level ("608 LL"), 8081 Low Level ("8081 LL"), and 8082 Low Level ("8082 LL), sample, label one 2mL graduated amber vials with Sample number and Batch number. No backup vial is created for this test.

10.3.25.1.2 For Methods 608, 8081, and 8082, sample, label two 2mL graduated amber vials with Sample number and Batch number. One for analysis and one for backup.

10.3.25.1.3 For "Shared Batch" 608 Low Level ("608 LL"), "Shared Batch" 8081 Low Level ("8081 LL"), and "Shared Batch" 8082 Low Level ("8082 LL) sample. Label two 2mL graduated amber vials with Sample number and Batch number. One for PCB analysis and one for pesticide analysis. No backup vial is created for this test.

10.3.25.1.4 For "Shared Batch" sample. Label three 2mL graduated amber vials with Sample number and Batch number. One for PCB analysis, one for pesticide analysis, and one for backup.

10.3.25.2 Bring sample to final volume. Top up and vial each sample individually. This prevents evaporation of solvent and concentration of sample.

10.3.25.2.1 For all Low-Level samples (608 LL, 8081 LL, 8082 LL, Shared Batch LL), final volume is 1mL.

10.3.25.2.2 For method 608, bring to 5mL volume by adding additional 4mL of hexane using a graduated cylinder. Mix using the vortex mixer for 10 seconds minimum.

10.3.25.2.3 For Methods 8081, 8082, and "Shared Batch", bring final volume to 10mL by adding additional 9mL of Hexane using a graduated cylinder. Mix using the vortex mixer for 10 seconds minimum.

10.3.25.3 Vialing Backup. For Methods 608, 8081, 8082, and "Shared Batch", vial 2mL into 2mL amber vial as a backup and do not add copper. Backup samples for pesticide and PCB are vialed prior to appropriate cleanup.

10.3.25.4 Vialing Sample.

10.3.25.4.1 For method 608 and 608 LL, place 1mL into 2mL amber vial and copper cleanup following EOP011.

10.3.25.4.2 For Methods 8081, 8082, 8081 LL, 8082 LL, place 1mL into 2mL amber vial and copper cleanup following EOP011.

10.3.25.4.3 For "Shared Batch", vial two separate 1mL aliquots of extract into 2mL amber vials and copper cleanup both following EOP011. For "Shared Batch LL", vial two separate 0.5mL aliquots of extract into 2mL amber vials and copper cleanup both following EOP011. One for Pesticide and one for PCB.

10.3.25.5 Discard unvialed remainder of the extract into hexane wastes.

10.3.25.6 Protect the extracts from light and store them in the designated freezer at -10°C.

10.3.26 Upon completion of extraction and vialing, account for all samples by matching to logbook entry and confirm the following information is complete and present. This information must be noted in real-time and not after completion.

10.3.26.1 Method. Start date and time, stop date and time, and method used
10.3.26.2 Sample. Matrix type and weights
10.3.26.3 Extract. Colors, final volumes, and cleanup procedures
10.3.26.4 Reagent. Reagents used, lot numbers, and volumes used
10.3.26.5 Spikes and surrogates. Lot numbers, concentration, and volumes used
10.3.26.6 Equipment. Equipment used, flow rates, and temperatures
10.3.26.7 Personnel. Document all personnel that have assisted in the extraction of the batch by having that person initial the logbook.
Note: It is not permissible under any circumstances to have another person initial for another person or take credit for work not performed.

10.3.27 In LIMS program, update status of samples to "DONE", using associated batch number, date of extraction, department, select samples being updated using "X", and saving changes. Document completion of sample extraction in Internal Chain program by changing status of batch to "OPEXT".

10.3.28 Present samples and logbook to team leads, supervisors, or managers for final review.

10.3.29 Deliver samples for analysis. Protocols and expectations for delivery are dictated by analytical department and are expected to be adhered to.

10.4 Acid-Base Neutrals by Methods EPA 625 & SW846 8270 (Sep Funnel, Water Bath, & Microsnyder or N-VAP).

Note: The sample preparation procedure for aqueous samples contains two options: standard volume extraction (1000ml) and reduced volume extraction (250ml) for large volume injection (LVI) analysis.

10.4.1 Confirm all samples present and delivered, listed on the LIMS batch sheet, and listed on preparation log. If any discrepancies, check with schedulers, team leads, supervisors, or managers.

10.4.2 If no discrepancies, document custody of samples in Internal Chain program and change status of batch to "ASN" reflecting the initiation of extraction in LIMS.

10.4.3 Label all apparatuses with the correct sample identification. Sample labels may transfer from one apparatus to the next.

10.4.4 Measure two 1-liter (or 250-ml for 3510C LVI) aliquots of deionized water for a method blank and blank spike into separate 1L or 250 ml amber glass bottles.

10.4.5 Mark level of sample on outside of glass bottle. For samples filled to completion, a mark is placed on the cap.

10.4.6 Mix the sample thoroughly by shaking.

Note: Samples which do not homogenize, are multiphasic, have a gasoline or oil odor, thick aqueous sludges, visible oil layer, or gels may not be appropriate for this extraction. These may require a reduced sample amount, alternate extraction technique, and comments to be added. Check with the team leads, supervisors, or managers for further instructions.

10.4.7 After mixing the sample, decant ¾" to 1" of sample into respective 2L separatory funnel. This is to allow for the addition of surrogate and/or spike.

10.4.8 Record the bottle number, sample description, and pH and chlorine (if needed) in the logbook.

10.4.9 Spiking & Surrogating. For proper execution protocols refer to EOP027.

10.4.9.1 Spiking Amounts. The test being requested will dictate the spikes that will need to be added. Using a calibrated 1mL syringe, add spike to blank spike, matrix spike, and matrix spike duplicate (if present).

10.4.9.1.1 For Base-Neutral ("BN") add 1.0mL (or 0.2ml for 3510C LVI) of each of the base neutral target compound spikes: BN1, BN2, & Aniline.

10.4.9.1.2 For Acid ("A") add 1.0mL (or 0.2ml for 3510C LVI) of the acid target compound spike.

10.4.9.1.3 For Acid Base-Neutral ("ABN") add 1.0mL (or 0.2ml for 3510C LVI) each of the Base-Neutral (BN1, BN2, Aniline) and Acid target compound spikes.

10.4.9.1.4 For SIM ("BS12SIM"), add 0.1 mL of acid target compound spike and 1.0 mL of Base-Neutral SIM ("BSIM") target compound spike.

Note: Check the expiration date for all spiking materials to verify that the solution is valid. Do not use expired solutions for spiking.

Note: Other special spikes may be needed. Check the special spike section of the logbook for additional information.

10.4.9.2 Surrogate Amounts. Add 1.0mL of the acid-base neutral surrogate using a calibrated 1mL syringe (or add 0.2ml for 3510C LVI) to all samples, blanks, and spikes. For SIM 1,4-Dioxane samples, add 1.0mL of 1,4-Dioxane-d8 solution.

Note: Check the expiration date for all surrogate materials to verify that the solution is valid. Do not use expired solutions for surrogating.

Note: Other special surrogates maybe needed. Check the logbook for additional information.

10.4.10 Pour samples into their respective 2L separatory funnel.

10.4.11 Rinse the sample bottle with 60 mL (or 15ml for 3510C LVI) of methylene chloride. Transfer the 60 mL methylene chloride rinse from the sample container into the separatory funnel containing the sample.

10.4.12 To determine the initial sample volume in the sample bottle, fill the empty sample jar with tap water to the mark on the jar. Pour the contents of the jar into a graduated cylinder and measure the volume. This is the sample volume that is recorded on the extraction log.

10.4.13 pH adjustment. pH will need to be adjusted based upon analysis type requested. Acid extraction (if requested) will take precedent over Base-Neutral extraction. If Acid extraction is not requested, proceed directly to Base-Neutral extraction.

10.4.13.1 For Acid only, Acid Base-Neutral, or SIM Samples. Adjust the pH of the sample to <2 using 1mL of the 50% sulfuric acid solution. Check the pH of the sample with pH test paper to verify the correct pH, adding more acid if needed, and document amount added in the extraction logbook.

10.4.13.1.1 Hand shake the funnel vigorously for 15-20 seconds. Release excess pressure by venting the funnel into the hood by opening and closing the Teflon stopcock.

10.4.13.1.2 Extract the sample by shaking the funnel vigorously for 2 minutes either using the automatic shaker or by hand. Allow the aqueous solvent layers to separate for a minimum of 10 minutes.

10.4.13.1.3 Collect the methylene chloride layer (the bottom layer) by passing through the drying column into the assembled Kuderna-Danish.

Note: Additional sodium sulfate may be added as needed to prevent trace amounts of aqueous fraction present in methylene chloride layer from passing through.

Important: Do not allow the sodium sulfate to become dry between solvent extracts.

10.4.13.1.4 Add a second 60-ml volume (or 15ml for 3510C LVI) of methylene chloride to the separatory funnel. Seal and shake the separatory funnel and repeat the extraction procedure described in Section 10.4.11.1.2 to 10.4.11.1.3. Perform a third extraction in the same manner.

10.4.13.1.5 If needed, once all three 60mL volumes (or 15ml for 3510C LVI) of methylene chloride have been added, centrifuge the solvent layer to break emulsions.

10.4.13.2 For Base-Neutral only, Acid Base-Neutral, BSIM, or SIM Samples. Adjust the pH to >11 with 4mL of the 10N sodium hydroxide solution, Check the pH of the sample with pH test paper to verify the correct pH, adding more base if needed, and document amount added in the extraction logbook. Add in 60mL of methylene chloride and repeat the shaking technique described in Sections 10.4.13.1.1 through 10.4.13.1.5.

Note: For Acid Base-Neutral or SIM Samples, pass the Base-Neutral extractions through the drying column and Kuderna-Danish setup utilized in the Acid extractions.

10.4.14 Once the three extractions or six extractions are completed, perform a final rinse of the drying column with an additional 10 mL of methylene Chloride. Allow the methylene chloride to drain through until dripping stops.

10.4.15 Remove drying column and discard sodium sulfate, glass wool or filter paper into solid sulfate waste.

10.4.16 Add in boiling chip and Snyder column. The sample is now ready to be concentrated.

10.4.17 Sample Concentration Procedure. Document the procedure in the extraction logbook.

10.4.17.1 Water Bath with Evaporation Collection System

Note: Fill in waterbath information, chiller information, and flow rates once samples have been loaded.

Note: If a sample ruptures into the waterbath, immediately close the hood, allow the boiling action of the water to subside, fumes to dissipate, and notify a team lead, supervisor, or manager. Do not attempt glassware removal or cleanup by yourself.

10.4.17.1.1 Position K-D flask in the waterbath. Immersing the 10mL concentrator tube into the waterbath. Concentrate samples to about 5-7mL.

10.4.17.1.2 Allow the K-D flask, receiver, and Snyder column to cool. During this process, approximately 1mL of methylene chloride will recondense and

flow down the side of the K-D flask removing most of the extract remaining in the flask and joints.

10.4.17.1.3 Remove Snyder column and slowly add 0.5-1mL of methylene chloride to the tilted K-D flask while rotate the flask. This is a final rinse of the flask and joint. Tilt upright to allow the methylene chloride to drain into the receiver.

10.4.17.1.4 Disassemble the K-D flask from the receiver.

10.4.18 Final volume adjustment. Samples may require further concentration (using the N-VAP or Micro Snyder methods) or addition to reach the desired final volume.

10.4.18.1 For Methods 8270, 625, and SIM, final volume is 1mL. For LVI extractions blow the extract directly down to a final volume of 2 ml on the N-Evap. If samples will not concentrate to final volumes or change states (i.e. liquid to gel), notify team lead, supervisors, and managers for further instructions. These may require a modified sample amount, alternate concentration technique, and comments to be added.

10.4.18.2 N-VAP Concentration Method. Immerse the nipple of the Buchi tube in the waterbath of the N-VAP. Direct a gentle stream of nitrogen gas onto the extract by adjusting height of needle until small dimple appears on surface. Evaporate the solvent until the desired volume is reached. Occasionally rinse the sidewalls of the concentrator tubes with methylene chloride during the evaporation step.

10.4.18.3 Micro Snyder Concentration Method. Attach a microsnyder column to the 10mL concentrator tube and immerse into the waterbath. Concentrate to the desired volume.

10.4.19 Vialing

10.4.19.1 For each sample, label a 2mL graduated amber vial with Sample number and Batch number. No backup vial is created for this test.

10.4.19.2 Bring final volume to 1mL with methylene chloride in receiver and mix with a Pasteur pipet. Transfer all sample extract to the vial.

10.4.19.3 Adjust the vial volume to 1mL with methylene chloride.

10.4.19.4 Protect the extracts from light and store them in the designated freezer at -10°C.

10.4.20 Upon completion of extraction and vialing, account for all samples by matching to logbook entry and confirm the following information is complete and present. This information must be noted in real-time and not after completion.

10.4.20.1 Method. Start date and time, stop date and time, and method used
10.4.20.2 Sample. Matrix type and weights
10.4.20.3 Extract. Colors, final volumes, and cleanup procedures
10.4.20.4 Reagent. Reagents used, lot numbers, and volumes used
10.4.20.5 Spikes and surrogates. Lot numbers, concentration, and volumes used

10.4.20.6 Equipment. Equipment used, flow rates, and temperatures.

10.4.20.7 Personnel. Document all personnel that have assisted in the extraction of the batch by having that person initial the logbook.

Note: It is not permissible under any circumstances to have another person initial for another person or take credit for work not performed.

10.4.21 In LIMS program, update status of samples to "DONE", using associated batch number, date of extraction, department, select samples being updated using "X", and saving changes. Document completion of sample extraction in Internal Chain program by changing status of batch to "OPEXT".

10.4.22 Present samples and logbook to team leads, supervisors, or managers for final review.

10.4.23 Deliver samples for analysis. Protocols and expectations for delivery are dictated by analytical department and are expected to be adhered to.

10.5 DRO - Diesel Range Organics for method SW846-8015C (Sep Funnel, Water Bath, & Microsnyder or N-VAP).

10.5.1 Confirm all samples present and delivered, listed on the LIMS batch sheet, and listed on preparation log. If any discrepancies, check with schedulers, team leads, supervisors, or managers.

10.5.2 If no discrepancies, document custody of samples in Internal Chain program and change status of batch to "ASN" reflecting the initiation of extraction in LIMS.

10.5.3 Label all apparatuses with the correct sample identification. Sample labels may transfer from one apparatus to the next.

10.5.4 Measure two 1-liter aliquots of deionized water for a method blank and blank spike into separate 1L amber glass bottles. For fingerprint ("FING") samples, only one method blank is required.

10.5.5 Add 1mL of HCl to blanks to match preservation method of the samples.

10.5.6 Mark level of sample on outside of glass bottle. For samples filled to completion, a mark is placed on the cap.

10.5.7 Mix the sample thoroughly by shaking.

Note: Samples which do not homogenize, are multiphasic, have a gasoline or oil odor, thick aqueous sludges, visible oil layer, or gels may not be appropriate for this extraction. These may require a reduced sample amount, alternate extraction technique, and comments to be added. Check with the team leads, supervisors, or managers for further instructions.

10.5.8 After mixing the sample, decant ¾" to 1" of sample into respective 2L separatory funnel. This is to allow for the addition of surrogate and/or spike.

10.5.9 Record the bottle number, sample description, and pH and chlorine (if needed) in the logbook.

10.5.10 Spiking & Surrogating. For proper execution protocols refer to EOP027.

10.5.10.1 Spiking Amounts. The test being requested will dictate the spikes that will need to be added. Add spike to blank spike, matrix spike, and matrix spike duplicate (if present).

10.5.10.1.1 Add 1mL of the DRO target compound spike using a calibrated 1mL syringe to the samples selected for matrix spikes.

10.5.10.1.2 "FING" samples do not have blank spikes, matrix spike, or matrix spike duplicates. Therefore, no target compound spike is necessary.

Note: Check the expiration date for all spiking materials to verify that the solution is valid. Do not use expired solutions for spiking.

10.5.10.2 Surrogate Amounts. Add 1mL of the DRO surrogate using a calibrated 1mL syringe to all samples, blanks, and spikes. "FING" samples require 4mL of DRO surrogate to be added using a calibrated 1mL syringe to all samples and method blank.

Note: Check the expiration date for all surrogating materials to verify that the solution is valid. Do not use expired solutions for surrogating.

10.5.11 Pour samples into their respective 2L separatory funnel.

10.5.12 Rinse the sample bottle with 60mL of methylene chloride. Transfer the 60mL methylene chloride rinse from the sample container into the separatory funnel containing the sample.

10.5.13 To determine the initial sample volume in the sample bottle, fill the empty sample jar with tap water to the mark on the jar. Pour the contents of the jar into a graduated cylinder and measure the volume. This is the sample volume that is recorded on the extraction log.

10.5.14 Hand shake the funnel vigorously for 15-20 seconds. Release excess pressure by venting the funnel into the hood by opening and closing the Teflon stopcock.

10.5.15 Extract the sample by shaking the funnel vigorously for 2 minutes either using the automatic shaker or by hand. Allow the aqueous solvent layers to separate for a minimum of 10 minutes.

10.5.16 Collect the methylene chloride layer (the bottom layer) by passing through the drying column into the assembled Kuderna-Danish.

Note: Additional sodium sulfate may be added as needed to prevent trace amounts of aqueous fraction present in methylene chloride layer from passing through.

Important: Do not allow the sodium sulfate to become dry between solvent extracts.

10.5.17 Add a second 60mL volume of methylene chloride to the separatory funnel. Seal and shake the separatory funnel and repeat the extraction procedure described in Section 10.5.13 to 10.5.14. Perform a third extraction in the same manner.

10.5.18 If needed, once all three 60mL volumes of methylene chloride have been added, centrifuge the solvent layer to break emulsions.

10.5.19 Once the three extraction steps are completed, perform a final rinse of the drying column with an additional 10mL of methylene chloride. Allow the methylene chloride to drain through until dripping stops.

10.5.20 Remove drying column and discard sodium sulfate, glass wool or filter paper into solid sulfate waste.

10.5.21 Add in boiling chip and Snyder column. The sample is now ready to be concentrated.

10.5.22 Sample Concentration Procedure. Document the procedure in the extraction logbook.

10.5.22.1 Water Bath with Evaporation Collection System

Note: Fill in waterbath information, chiller information, and flow rates once samples have been loaded.

Note: If a sample ruptures into the waterbath, immediately close the hood, allow the boiling action of the water to subside, fumes to dissipate, and notify a team lead, supervisor, or manager. Do not attempt glassware removal or cleanup by yourself.

10.5.22.1.1 Position K-D flask in the waterbath. Immersing the 10mL concentrator tube into the waterbath. Concentrate samples to about 5-7mL.

10.5.22.1.2 Allow the K-D flask, receiver, and Snyder column to cool. During this process, approximately 1mL of methylene chloride will recondense and flow down the side of the K-D flask removing most of the extract remaining in the flask and joints.

10.5.22.1.3 Remove Snyder column and slowly add 0.5-1mL of methylene chloride to the tilted K-D flask while rotate the flask. This is a final rinse of the flask and joint. Tilt upright to allow the methylene chloride to drain into the receiver.

10.5.22.2 Disassemble the K-D flask from the receiver.

10.5.23 Final volume adjustment. Samples may require further concentration using the N-VAP or Micro Snyder methods to reach the desired final volume.

10.5.23.1 For Methods 8015 and “FING”, final volume is 1mL. If samples will not concentrate to final volumes or change states (i.e. liquid to gel), notify team lead, supervisors, and managers for further instructions. These may require a modified sample amount, alternate concentration technique, and comments to be added.

10.5.23.2 N-VAP Method. Immerse the nipple of the receiver in the waterbath of the N-VAP. Direct a gentle stream of nitrogen gas onto the extract by adjusting height of needle until small dimple appears on surface. Evaporate the solvent until the desired volume is reached. Occasionally rinse the sidewalls of the concentrator tubes with methylene chloride during the evaporation step.

10.5.23.3 Micro Snyder Method. Attach a micro-snyder column to the receiver and immerse into a waterbath. Concentrate to the desired volume.

10.5.24 Vialing

10.5.24.1 For each sample, label a 2mL graduated amber vial with Sample number and Batch number. No backup vial is created for this test.

10.5.24.2 Bring final volume to 1mL with methylene chloride in receiver and mix with a Pasteur pipet. Transfer all sample extract to the vial.

10.5.24.3 Adjust the vial volume to 1mL with methylene chloride.

10.5.24.4 Protect the extracts from light and store them in the designated freezer at -10°C.

10.5.25 Upon completion of extraction and vialing, account for all samples by matching to logbook entry and confirm the following information is complete and present. This information must be noted in real-time and not after completion.

10.5.25.1 Method. Start date and time, stop date and time, and method used
10.5.25.2 Sample. Matrix type and weights
10.5.25.3 Extract. Colors, final volumes, and cleanup procedures
10.5.25.4 Reagent. Reagents used, lot numbers, and volumes used
10.5.25.5 Spikes and surrogates. Lot numbers, concentration, and volumes used
10.5.25.6 Equipment. Equipment used, flow rates, and temperatures.
10.5.25.7 Personnel. Document all personnel that have assisted in the extraction of the batch by having that person initial the logbook.

Note: It is not permissible under any circumstances to have another person initial for another person or take credit for work not performed.

10.5.26 In LIMS program, update status of samples to “DONE”, using associated batch number, date of extraction, department, select samples being updated using “X”, and saving changes. Document completion of sample extraction in Internal Chain program by changing status of batch to “OPEXT”.

10.5.27 Present samples and logbook to team leads, supervisors, or managers for final review.

10.5.28 Deliver samples for analysis. Protocols and expectations for delivery are dictated by analytical department and are expected to be adhered to.

10.6 NJ EPH- Extractable Petroleum Hydrocarbons (Sep Funnel, Water Bath, & Micro Snyder or N-VAP).

10.6.1 Confirm all samples present and delivered, listed on the LIMS batch sheet, and listed on preparation log. If any discrepancies, check with schedulers, team leads, supervisors, or managers.

10.6.2 If no discrepancies, document custody of samples in Internal Chain program and change status of batch to "ASN" reflecting the initiation of extraction in LIMS.

10.6.3 Label all apparatuses with the correct sample identification. Sample labels may transfer from one apparatus to the next.

10.6.4 Measure three 1-liter aliquots of deionized water for a method blank, blank spike (BS1), and second blank spike (labeled as "BS11") into separate 1L amber glass bottles.

10.6.5 Add 1mL of HCl to blanks to match preservation method of the samples.

10.6.6 Mark level of sample on outside of glass bottle. For samples filled to completion, a mark is placed on the cap.

10.6.7 Mix the sample thoroughly by shaking.

Note: Samples which do not homogenize, are multiphasic, have a gasoline or oil odor, thick aqueous sludges, visible oil layer, or gels may not be appropriate for this extraction. These may require a reduced sample amount, alternate extraction technique, and comments to be added. Check with the team leads, supervisors, or managers for further instructions.

10.6.8 After mixing the sample, decant ¾" to 1" of sample into respective 2L separatory funnel. This is to allow for the addition of surrogate and/or spike.

10.6.9 Record the bottle number, sample description, and pH and chlorine (if needed) in the logbook.

10.6.10 Spiking & Surrogating. For proper execution protocols refer to EOP027.

10.6.10.1 Spiking Amounts. The test being requested will dictate the spikes that will need to be added. Add spike to blank spike, matrix spike, and matrix spike duplicate (if present).

Note: EPH spike in use must be stored at room temperature. If inadvertently placed into the refrigerator or freezer, remove and allow to return to room temperature.

10.6.10.1.1 Add 1mL of the EPH target compound spike using a calibrated 1mL syringe to the blank spike (BS1), matrix spike (MS), matrix spike duplicate (MSD) and blank spike duplicate (BSD) if present.

10.6.10.1.2 Add 0.5mL of the DRO target compound spike using a calibrated 1mL syringe to the second blank spike (BS11).

Note: Check the expiration date for all spiking materials to verify that the solution is valid. Do not use expired solutions for spiking.

10.6.10.2 Surrogate Amounts. Add 1mL of the EPH surrogate using a calibrated 1mL syringe to all samples, blanks, and spikes.

Note: Check the expiration date for all surrogating materials to verify that the solution is valid. Do not use expired solutions for surrogating.

10.6.11 Pour samples into their respective 2L separatory funnel.

10.6.12 Rinse the sample bottle with 60mL of methylene chloride. Transfer the 60mL methylene chloride rinse from the sample container into the separatory funnel containing the sample.

10.6.13 To determine the initial sample volume in the sample bottle, fill the empty sample jar with tap water to the mark on the jar. Pour the contents of the jar into a graduated cylinder and measure the volume. This is the sample volume that is recorded on the extraction log.

10.6.14 Hand shake the funnel vigorously for 15-20 seconds. Release excess pressure by venting the funnel into the hood by opening and closing the Teflon stopcock.

10.6.15 Extract the sample by shaking the funnel vigorously for 2 minutes either using the automatic shaker or by hand. Allow the aqueous solvent layers to separate for a minimum of 10 minutes.

10.6.16 Collect the methylene chloride layer (the bottom layer) by passing through the drying column into the assembled Kuderna-Danish.

Note: Additional sodium sulfate may be added as needed to prevent trace amounts of aqueous fraction present in methylene chloride layer from passing through.

Important: Do not allow the sodium sulfate to become dry between solvent extracts.

10.6.17 Add a second 60mL volume of methylene chloride to the separatory funnel. Seal and shake the separatory funnel and repeat the extraction procedure described in Section 10.5.13 to 10.5.14. Perform a third extraction in the same manner.

10.6.18 If needed, once all three 60mL volumes of methylene chloride have been added, centrifuge the solvent layer to break emulsions.

10.6.19 Once the three extraction steps are completed, perform a final rinse of the drying column with an additional 10mL of methylene chloride. Allow the methylene chloride to drain through until dripping stops.

10.6.20 Remove drying column and discard sodium sulfate, glass wool or filter paper into solid sulfate waste.

10.6.21 Add in boiling chip and Snyder column. The sample is now ready to be concentrated.

10.6.22 Sample Concentration Procedure. Document the procedure in the extraction logbook.

10.6.22.1 Water Bath with Evaporation Collection System

Note: Fill in waterbath information, chiller information, and flow rates once samples have been loaded.

Note: If a sample ruptures into the waterbath, immediately close the hood, allow the boiling action of the water to subside, fumes to dissipate, and notify a team lead, supervisor, or manager. Do not attempt glassware removal or cleanup by yourself.

10.6.22.1.1 Position K-D flask in the waterbath. Immersing the 10mL concentrator tube into the waterbath. Concentrate samples to about 5-7mL.

10.6.22.1.2 Allow the K-D flask, receiver, and Snyder column to cool. During this process, approximately 1mL of methylene chloride will recondense and flow down the side of the K-D flask removing most of the extract remaining in the flask and joints.

10.6.22.1.3 Remove Snyder column and slowly add 0.5-1mL of methylene chloride to the tilted K-D flask while rotate the flask. This is a final rinse of the flask and joint. Tilt upright to allow the methylene chloride to drain into the receiver.

10.6.22.2 Disassemble the K-D flask from the receiver.

10.6.23 Final volume adjustment. Samples may require further concentration using the N-VAP or Micro Snyder methods to reach the desired final volume.

10.6.23.1 For NJ EPH final volume is 1mL. If samples will not concentrate to final volumes or change states (i.e. liquid to gel), notify team lead, supervisors, and managers for further instructions. These may require a modified sample amount, alternate concentration technique, and comments to be added.

10.6.23.2 N-VAP Method. Immerse the nipple of the receiver in the waterbath of the N-VAP. Direct a gentle stream of nitrogen gas onto the extract by adjusting height of needle until small dimple appears on surface. Evaporate the solvent until the desired volume is reached. Occasionally rinse the sidewalls of the concentrator tubes with methylene chloride during the evaporation step.

10.6.23.3 Micro Snyder Method. Attach a micro-snyder column to the receiver and immerse into a waterbath. Concentrate to the desired volume.

10.6.24 Vialing

10.6.24.1 For each sample, label a 2mL graduated amber vial with Sample number and Batch number. No backup vial is created for this test.

10.6.24.2 Bring final volume to 1mL with methylene chloride in receiver and mix with a Pasteur pipet. Transfer all sample extract to the vial.

10.6.24.3 Adjust the vial volume to 1mL with methylene chloride.

10.6.24.4 Protect the extracts from light and store them in the designated freezer at -10°C.

10.6.25 Upon completion of extraction and vialing, account for all samples by matching to logbook entry and confirm the following information is complete and present. This information must be noted in real-time and not after completion.

10.6.25.1 Method. Start date and time, stop date and time, and method used
10.6.25.2 Sample. Matrix type and weights
10.6.25.3 Extract. Colors, final volumes, and cleanup procedures
10.6.25.4 Reagent. Reagents used, lot numbers, and volumes used
10.6.25.5 Spikes and surrogates. Lot numbers, concentration, and volumes used
10.6.25.6 Equipment. Equipment used, flow rates, and temperatures.
10.6.25.7 Personnel. Document all personnel that have assisted in the extraction of the batch by having that person initial the logbook.
Note: It is not permissible under any circumstances to have another person initial for another person or take credit for work not performed.

10.6.26 In LIMS program, update status of samples to "DONE", using associated batch number, date of extraction, department, select samples being updated using "X", and saving changes. Document completion of sample extraction in Internal Chain program by changing status of batch to "OPEXT".

10.6.27 Present samples and logbook to team leads, supervisors, or managers for final review.

10.6.28 Deliver samples for analysis. Protocols and expectations for delivery are dictated by analytical department and are expected to be adhered to.

10.7 NJ OQA-QAM-025 (NJ Semi-VOA Petroleum Hydrocarbon) (Sep Funnel, Water Bath, & Micro Snyder or N-VAP).

10.7.1 Confirm all samples present and delivered, listed on the LIMS batch sheet, and listed on preparation log. If any discrepancies, check with schedulers, team leads, supervisors, or managers.

10.7.2 If no discrepancies, document custody of samples in Internal Chain program and change status of batch to "ASN" reflecting the initiation of extraction in LIMS.

10.7.3 Label all apparatuses with the correct sample identification. Sample labels may transfer from one apparatus to the next.

10.7.4 Measure two 1-liter aliquots of deionized water for a method blank and blank spike into separate 1L amber glass bottles.

10.7.5 Mark level of sample on outside of glass bottle. For samples filled to completion, a mark is placed on the cap.

10.7.6 Mix the sample thoroughly by shaking.

Note: Samples which do not homogenize, are multiphasic, have a gasoline or oil odor, thick aqueous sludges, visible oil layer, or gels may not be appropriate for this extraction.

These may require a reduced sample amount, alternate extraction technique, and comments to be added. Check with the team leads, supervisors, or managers for further instructions.

10.7.7 After mixing the sample, decant ¾" to 1" of sample into respective 2L separatory funnel. This is to allow for the addition of surrogate and/or spike.

10.7.8 Record the bottle number, sample description, and pH and chlorine (if needed) in the logbook.

10.7.9 Spiking & Surrogating. For proper execution protocols refer to EOP027.

10.7.9.1 Spiking Amounts. The test being requested will dictate the spikes that will need to be added. Using a calibrated 1mL syringe or 1 mL volumetric pipette, add spike to blank spike, matrix spike, and matrix spike duplicate (if present).

10.7.9.1.1 Add 1mL of the DRO target compound spike using a calibrated 1mL syringe to the samples selected for matrix spikes.

Note: Check the expiration date for all spiking materials to verify that the solution is valid. Do not use expired solutions for spiking.

Note: Other special spikes maybe needed. Check the special spike section of the logbook for additional information.

10.7.9.2 Surrogate Amounts. Add 1mL of the OQA surrogate using a calibrated 1mL syringe to all samples, blanks, and spikes.

Note: Check the expiration date for all surrogate materials to verify that the solution is valid. Do not use expired solutions for surrogating.

Note: Other special surrogates maybe needed. Check the logbook for additional information.

10.7.10 Pour samples into their respective 2L separatory funnel.

10.7.11 Rinse the sample bottle with 60mL of methylene chloride. Transfer the 60mL methylene chloride rinse from the sample container into the separatory funnel containing the sample.

10.7.12 To determine the initial sample volume in the sample bottle, fill the empty sample jar with tap water to the mark on the jar. Pour the contents of the jar into a graduated cylinder and measure the volume. This is the sample volume that is recorded on the extraction log.

10.7.13 Hand shake the funnel vigorously for 15-20 seconds. Release excess pressure by venting the funnel into the hood by opening and closing the Teflon stopcock.

10.7.14 Extract the sample by shaking the funnel vigorously for 2 minutes either using the automatic shaker or by hand. Allow the aqueous solvent layers to separate for a minimum of 10 minutes.

10.7.15 Collect the methylene chloride layer (the bottom layer) by passing through the drying column into the assembled Kuderna-Danish.

Note: Additional sodium sulfate may be added as needed to prevent trace amounts of aqueous fraction present in methylene chloride layer from passing through.

Important: Do not allow the sodium sulfate to become dry between solvent extracts.

10.7.16 Add a second 60mL volume of methylene chloride to the separatory funnel. Seal and shake the separatory funnel and repeat the extraction procedure described in Section 10.6.12 to 10.6.13. Perform a third extraction in the same manner.

10.7.17 If needed, once all three 60mL volumes of methylene chloride have been added, centrifuge the solvent layer to break emulsions.

10.7.18 Once the three extraction steps are completed, perform a final rinse of the drying column with an additional 10mL of methylene Chloride. Allow the methylene chloride to drain through until dripping stops.

10.7.19 Remove drying column and discard sodium sulfate, glass wool or filter paper into solid sulfate waste.

10.7.20 Add in boiling chip and Snyder column. The sample is now ready to be concentrated.

10.7.21 Sample Concentration Procedure. Document the procedure in the extraction logbook.

10.7.21.1 Water Bath with Evaporation Collection System

Note: Fill in waterbath information, chiller information, and flow rates once samples have been loaded.

Note: If a sample ruptures into the waterbath, immediately close the hood, allow the boiling action of the water to subside, fumes to dissipate, and notify a team lead, supervisor, or manager. Do not attempt glassware removal or cleanup by yourself.

10.7.21.1.1 Position K-D flask in the waterbath. Immersing the 10mL concentrator tube into the waterbath. Concentrate samples to about 5-7mL.

10.7.21.1.2 Allow the K-D flask, receiver, and Snyder column to cool. During this process, approximately 1mL of methylene chloride will recondense and flow down the side of the K-D flask removing most of the extract remaining in the flask and joints.

10.7.21.1.3 Remove Snyder column and slowly add 0.5-1mL of methylene chloride to the tilted K-D flask while rotate the flask. This is a final rinse of the flask and joint. Tilt upright to allow the methylene chloride to drain into the receiver.

10.7.21.2 Disassemble the K-D flask from the receiver.

10.7.22 Final volume adjustment. Samples may require further concentration using the N-VAP or Micro Snyder methods to reach the desired final volume.

10.7.22.1 For NJ OQA 25, final volume is 1mL. If samples will not concentrate to final volumes or if they change states (i.e. liquid to gel), notify team lead, supervisors, and managers for further instructions. These may require a modified sample amount, alternate concentration technique, and comments to be added.

10.7.22.2 N-VAP Method. Immerse the nipple of the receiver in the waterbath of the N-VAP. Direct a gentle stream of nitrogen gas onto the extract by adjusting height of needle until small dimple appears on surface. Evaporate the solvent until the desired volume is reached. Occasionally rinse the sidewalls of the concentrator tubes with methylene chloride during the evaporation step.

10.7.22.3 Micro Snyder Method. Attach a micro-snyder column to the receiver and immerse into a waterbath. Concentrate to the desired volume.

10.7.23 Vialing

10.7.23.1 For each sample, label a 2mL graduated amber vial with Sample number and Batch number. No backup vial is created for this test.

10.7.23.2 Bring final volume to 1mL with methylene chloride in receiver and mix with a Pasteur pipet. Transfer all sample extract to the vial.

10.7.23.3 Adjust the vial volume to 1mL with methylene chloride.

10.7.23.4 Protect the extracts from light and store them in the designated freezer at -10°C.

10.7.24 Upon completion of extraction and vialing, account for all samples by matching to logbook entry and confirm the following information is complete and present. This information must be noted in real-time and not after completion.

10.7.24.1 Method. Start date and time, stop date and time, and method used
10.7.24.2 Sample. Matrix type and weights
10.7.24.3 Extract. Colors, final volumes, and cleanup procedures
10.7.24.4 Reagent. Reagents used, lot numbers, and volumes used
10.7.24.5 Spikes and surrogates. Lot numbers, concentration, and volumes used
10.7.24.6 Equipment. Equipment used, flow rates, and temperatures.
10.7.24.7 Personnel. Document all personnel that have assisted in the extraction of the batch by having that person initial the logbook.
Note: It is not permissible under any circumstances to have another person initial for another person or take credit for work not performed.

10.7.25 In LIMS program, update status of samples to “DONE”, using associated batch number, date of extraction, department, select samples being updated using “X”, and saving changes. Document completion of sample extraction in Internal Chain program by changing status of batch to “OPEXT”.

10.7.26 Present samples and logbook to team leads, supervisors, or managers for final review.

10.7.27 Deliver samples for analysis. Protocols and expectations for delivery are dictated by analytical department and are expected to be adhered to.

11.0 CALCULATIONS

11.1 Not applicable.

12.0 QUALITY ASSURANCE

12.1 Extract a method blank and blank spike at a rate of one per day or every twenty (20) samples, whichever is more frequent.

12.2 A matrix spike / matrix spike duplicate (MS/MSD) is required per every 20 samples of similar matrix. If there is insufficient sample volume in the batch for MS/MSD pair, a BS/BSD will be used in place of a MS/MSD to evaluate accuracy and precision.

12.3 For pest/PCBs a separate MB, BSP, and MS/MSD set are needed if the sample requires PCBs, Toxaphene and/or chlordane. Therefore, if a sample is to be extracted for pesticides and PCBs, 2 sets of MB, BS, MS, and MSD are needed.

12.4 Refer to Project Specific Bench Notes (GC8081, GC8082, MS8270) for additional program or client specific QC requirements.

13.0 DOCUMENTATION

13.1 All sample preparation activities and related information must be documented in the respective extraction logbook. Complete all information required for the extraction summary logbook.

13.1.1 Errors must be stricken with a single line, initialed and dated. The correct information must be written adjacent to the erroneous information.

13.1.2 The individual with custody responsibility must sign the extraction log.

13.1.3 The individuals involved in the processing of the batch must initial in the appropriate respective locations.

13.1.4 All spikes, surrogates, reagents, solutions, and applicable manufacturer and lot information must be filled in the appropriate respective locations.

13.1.5 All equipment, equipment identifiers, and the operating parameters at time of use must be filled in the appropriate respective locations.

13.1.6 The logbooks and/or e-logs must be reviewed and initialed by a team leader or supervisor. The approved page is copied and given to the Report Generation Dept.

13.2 Sample custody must be updated according to proper Chain of Custody protocols.

13.3 All standards preparation must be documented in the standards preparation logbook.

13.4 Equipment maintenance logs must be maintained.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. Follow all safety practices that limit the escape of vapors, liquids, or solids to the environment. All method users must be familiar with the waste management practices described in section 14.2.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non-hazardous aqueous wastes.

14.2.2 Hazardous aqueous wastes

14.2.3 Chlorinated organic solvents

14.2.4 Non-chlorinated organic solvents

14.2.5 Hazardous solid wastes

14.2.6 Non-hazardous

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
12.2	Added BS/BSD when MS/MSD cannot be prepped

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
22	9/12/18	Thomas Gabriel
23	9/17/19	Olga Azarian
24	10/29/19	Wahied Bayoumi
25	8/13/20	Wahied Bayoumi
26	3/12/21	Adrian Lee
27	9/27/21	Olga Azarian

END OF DOCUMENT

LAB SUPERVISOR: Naisha Tow

QA OFFICER: Olga V. Agudelo

EFFECTIVE DATE: 9-20-2022

TITLE: MICROWAVE EXTRACTION PROCEDURE (BASE, NEUTRAL, ACID ORGANICS; PESTICIDES, POLYCHLORINATED BIPHENYLS, DIESEL RANGE ORGANICS, NEW JERSEY EXTRACTABLE PETROLEUM HYDROCARBONS)
REFERENCES: SW846 3546
REVISED SECTIONS: 10.3.12.4

1.0 SCOPE AND APPLICATION

1.1 This method describes the extraction procedure of semi-volatile organic compounds in various solid sources for analysis by gas chromatography (GC) or gas chromatography/mass spectrometry (GC/MS).

2.0 SUMMARY

2.1 An aliquot of solid sample such as soils, sludges, and wastes, is extracted with an organic solvent (methylene chloride or hexane) utilizing the microwave extraction process. The extract is concentrated and/or solvent exchanged for instrumental analysis techniques using SW-846 Methods 8081B, 8082A, 8270E, and 8015D.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Not Applicable. See determinative method.

4.0 DEFINITIONS

BLANK. An analytical sample designed to assess specific sources of laboratory contamination. Types of Blanks may include: Method Blank, Instrument Blank, Storage Blank, and Leachate Blank.

BATCH. A group of samples and associated quality control samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

BLANK SPIKE (BS). An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

CLASS A GLASSWARE. Volumetric laboratory glass that has been manufactured, calibrated and certified to established ASTM volume standards. Under normal laboratory conditions, Class A Glassware does not require volume calibration or verification.

EXTRACTION. The process of removing a desired compound from a matrix using chemical or mechanical procedures. The process is used to isolate and concentrate targeted constituents for instrumental analysis

GAS LIQUID CHROMATOGRAPHY (GC). An instrumental procedure used to separate mixtures of organic compounds based on polarity or boiling point. The technique employs long capillary columns containing a high molecular weight organic polymer to effect the separation.

HOLDING TIME. The maximum time that samples may be held prior to preparation and/or analysis and still are considered valid.

KUDERNA DANISH. A three-stage glass solvent concentration device consisting of a large volume receiving flask (250 or 500mL), a small volume receiver and a three-ball air cooled condenser (Snyder column). This device is used to evaporate large volumes of solvent used for organic extractions to increase the concentration of the analyte in the solvent.

MATRIX. The predominant material of which a sample is composed. For the purpose of this method, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX SPIKE. Aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE. A second aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

METHOD BLANK. An analytical control consisting of all reagents, internal standards, and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

REAGENT WATER. Purified water in which an interferant is not observed at or above the minimum reporting limit of the parameters of interest.

SURROGATE. An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Laboratory Safety Manual and SGS Safety Policy, which includes the use of safety glasses and lab coats. Handle all acids, which are corrosive with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, treat each chemical as a potential health hazard. Reduce exposure to these reagents to the lowest possible level. The laboratory is responsible for maintaining a current file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data sheets is available to all personnel involved in these analyses.

6.0 COLLECTION, PRESERVATION, & HOLDING TIMES

6.1 Collect solid samples in 8-32oz. glass jars.

6.2 Collect wipe samples in 4-8oz. widemouth glass jars.

6.3 10 grams to approximately 12 grams (for DRO), 15 grams to approximately 17 grams (for Pesticides, PCBs, and EPH), or 30 grams to approximately 33 grams (for ABNs) of solid samples are required for an extraction. Additional sample volume may be necessary for any samples used for matrix duplicates and matrix spikes.

6.4 The samples must be protected from light and refrigerated at <6°C from the time of receipt until extraction and analysis.

6.5 Extract samples within 14 days of sampling and analyze the extract within 40 days of the extraction.

7.0 APPARATUS & MATERIALS

7.1 Widemouth Glass Bottle- 150mL or 250mL

7.2 Stainless Steel Spoon or Wooden Spatula

7.3 Top Loading balance, capable of weighing to 0.01g

7.4 Class A Volumetric Flasks: 1mL, 5mL , 10mL, 100mL volumes

7.5 One (1) mL volumetric pipettes or pre-calibrated 1mL syringes

7.6 Microwave Extractor- for sample extraction. Models used include (but are not limited to): Mars-xpress, Mar 5, or Mar 6

7.7 Microwave extraction vessels. Composed of Teflon tube with Teflon pressure cap (Composed of pressure plug and cap)

7.8 Drying column – used for removing aqueous fraction from organic fraction. Composed of glass funnel (large or small) containing glass wool or filter paper and sodium sulfate

7.9 Buchi Concentrator System- solvent concentrator capable of capturing evaporated solvent. Composed of Syncore Unit, Vacuum Pump, Chiller, & Recirculating Pump

7.10 Buchi tube, Class A- for Buchi concentrator system.

7.11 N-EVAP Concentrator- small volume concentrator. Composed of waterbath and Nitrogen gas blow down apparatus.

7.12 2mL graduated amber glass vial and associated PTFE lined cap (screw top or crimp)

7.13 Refrigerator & Freezer- for sample and backup storage

7.14 10 ml graduated cylinder

7.15 Vortex Mixer

8.0 STANDARDS & REAGENTS

8.1 Reagent water - deionized and carbon filtered water prepared to ASTM Type II specifications.

8.2 Solvents - reagent grade for trace organic analysis. Each solvent lot must be checked for interferences prior to use. Refer to SOP EOP013 for the procedure regarding solvent lot approval.

8.2.1 Methylene Chloride

8.2.2 Hexane

8.2.3 Acetone

8.3 Solutions- generated from reagent grade for trace organic analysis solvents listed above or shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.

8.3.1 1:1 Methylene Chloride:Acetone- Mix equal parts Methylene Chloride and Acetone.

8.3.2 2:1 Hexane:Acetone- Mix two parts Hexane with one part Acetone.

8.3.3 Sulfuric acid (1:1)- purchase as a commercially prepared solution (50% v/v). Alternatively prepare a 50% solution from equal parts concentrated sulfuric acid and reagent water

8.3.4 10N NaOH purchased commercially.

8.4 Baked Hydromatrix- refer to EOP029 for preparation instructions.

8.5 Baked Sodium sulfate - granular anhydrous, refer to EOP029 for preparation instructions

8.6 Nitrogen Gas – High Purity Grade.

8.7 Surrogate and Matrix Spiking Solutions. See the analytical SOP for target compound specifications. See the extraction SOP for target compound amounts. Refer to the most current version of Form: OP026 for details on Standard and Spike preparation.

Note: Unopened stock solutions must be stored according to the manufacturers documented holding time and storage temperature recommendations. After opening, stock standards must be replaced after 6 months or sooner if manufacturer's expiration date comes first or comparison with quality control check samples indicate degradation.

9.0 INTERFERENCES

9.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. Demonstrate that these materials are free from interferences under the conditions of the analysis by analyzing method blanks.

9.2 Interferences co-extracted from the samples will vary considerably from source to source. If interferences prevent the analysis of an extracted sample, further cleanup of the sample extract may be necessary.

9.3 Phthalate esters contaminate many products commonly used in the laboratory. Avoid plastics in particular because they contain phthalates, used as plasticizers, which can leach from these materials. Practice sound, consistent materials control to avoid phthalate contamination, which may occur at any time.

9.4 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Specifically, Aldrin, Heptachlor, and most organophosphorus pesticides will degrade in this situation. This occurs in glassware that is difficult to rinse. Carefully hand-rinse these items to avoid this problem.

10.0 PROCEDURE

10.1 Assemble all needed apparatuses and rinse prior to use.

10.1.1 Microwave Extractor Tubes

10.1.1.1 Microwave Tubes. Prepare previously washed microwave tubes by adding 5-10mL of DCM and shaking/swirling until all inner surfaces have come in contact with the DCM. Discard methylene chloride by pouring off into chlorinated solvent waste container.

10.1.1.1.1 If water is present, rinse with 5-10mL of acetone prior to methylene chloride prep.

10.1.1.1.2 Hexane rinse, if needed, may also be performed after the methylene chloride rinse.

10.1.1.2 Pressure Plugs. Prepare previously washed pressure plugs by rinsing with DCM and shaking/swirling until all surfaces have come in contact with the DCM. Discard methylene chloride by pouring off into chlorinated solvent waste container.

10.1.1.2.1 If water is present, rinse with 5-10mL of acetone prior to methylene chloride prep.

10.1.1.2.2 Hexane rinse, if needed, may also be performed after the methylene chloride rinse.

10.1.2 Drying Column Preparation

10.1.2.1 Prepare a drying column by adding approximately 15-20g of baked sodium sulfate into a filter paper funnel.

10.1.3 Buchi Tube Sample Collection Setup Preparation

10.1.3.1 Prepare previously washed and baked dry Buchi Tubes by adding 5-10mL of DCM and shaking/swirling until all inner surfaces have come in contact with the DCM. Discard methylene chloride by pouring off into chlorinated solvent waste container.

10.1.3.1.1 If water is present, rinse with 5-10mL of acetone prior to methylene chloride prep.

10.1.3.1.2 Hexane rinse, if needed, may also be performed after the methylene chloride rinse.

10.1.3.2 Place a drying column on top of Buchi tube to complete Buchi Tube Sample Collection Setup.

10.2 Turn on all equipment, check for proper operating conditions, and allow to reach operating temperatures prior to use.

10.2.1 Microwave Extractor- models used include (but are not limited to): Ethos Ex Microwave Extraction System, Mars-xpress, or Mar 6.

10.2.1.1 Inspect inside and outside of unit for damage. If any damage is noted, notify team lead, supervisor, or manager.

10.2.1.2 Turn Microwave Extractor on. If unit will not turn on, notify team lead, supervisor, or manager.

10.2.1.3 Allow unit to warm up.

10.2.1.4 Inspect carousel, microwave tubes, pressure plug, and caps for damage. If any damage is noted, notify team lead, supervisor, or manager.

10.2.2 Waterbath with Evaporation Collection System. System is composed of 2 parts: Waterbath and Condensation System (Chiller and Condensers). Each part will need to be checked prior to use.

10.2.2.1 Chiller

10.2.2.1.1 Check coolant level in chiller and top-up as needed.

10.2.2.1.2 Turn on chiller. Temperature of coolant will show up on digital display, and flowmeter will register rate of coolant flow in gallons per minute (gpm).

10.2.2.1.3 Check coolant lines for leaks. If any are noted, turn off chiller and notify team lead, supervisor, or manager.

10.2.2.1.4 Adjust flowrate of chiller to exceed flow rate listed on chiller operating parameter card.

10.2.2.1.5 Monitor coolant temperature and ensure it falls below the maximum chiller temperature listed on chiller operating parameter card. If it will not fall below maximum temperature, notify team lead, supervisor, or manager.

10.2.2.1.6 Continue to monitor coolant temperature throughout use. At any point of operation, if chiller temperature exceeds listed maximum, remove all samples and suspend use of the associated waterbath(s) until temperature falls below maximum. If it doesn't fall below maximum temperature, notify team lead, supervisor, or manager.

10.2.2.2 Waterbath

10.2.2.2.1 Check water level in waterbath. If water level is below ¾" of sample holding deck, top up with water.

10.2.2.2.2 Turn on waterbath. If waterbath will not turn on, notify team lead, supervisor, or manager.

10.2.2.2.3 Temperature of water in waterbath will show up on digital display. Check and adjust to ensure temperature is set to 75.0±5.0°C. Water set point temperature is never allowed to exceed 75.0°C. Water temperature is never allowed to exceed 80.0°C. Calibration of waterbath temperature occurs on a quarterly basis. Check calibration date- if expired, do not use. If waterbath will not turn on or out of calibration, notify team lead, supervisor, or manager.

10.2.2.2.4 Continue to monitor water temperature throughout use to ensure heating elements are operating normally and maintain water set point temperature. If waterbath does not maintain the water set point temperature, notify team lead, supervisor, or manager.

10.2.3 Buchi Concentrator System. System is composed of 2 parts: Buchi and Condensation System (Chiller and Condenser). Each part will need to be checked prior to use.

10.2.3.1 Refer to OP030 for proper Buchi Concentrator System Start-up instructions.

10.2.3.2 Refer to Table 3 for programs, steps, pressure settings, and run times.

10.2.3.3 If any issues, notify a team lead, supervisor, or manager.

10.2.4 N-EVAP Concentrator. System is composed of 2 parts: Waterbath and Nitrogen Flow System. Each part will need to be checked prior to use.

10.2.4.1 Waterbath

10.2.4.1.1 Check water level in waterbath. If water level is below ¾" of top, top up with water.

10.2.4.1.2 Turn on waterbath. If waterbath will not turn on, notify team lead, supervisor, or manager.

10.2.4.1.3 Digital Display Waterbath. Temperature of water in waterbath will show up on digital display. Check and adjust to ensure temperature is set to 35.0±2.0°C. Water set point temperature is never allowed to exceed 37.0°C. If waterbath will not turn on, notify team lead, supervisor, or manager.

10.2.4.1.4 Manual Knob Waterbath. Temperature of water in waterbath will need to be manually calibrated and proper setting noted. Check and adjust to ensure temperature is set to 35.0±2.0°C. Water temperature is never allowed to exceed 37.0°C. If waterbath will not turn on or maintain proper temperature, notify team lead, supervisor, or manager.

10.2.4.1.5 Continue to monitor water temperature throughout use to ensure heating elements are operating normally and maintain water set point temperature. If waterbath does not maintain the water set point temperature, notify team lead, supervisor, or manager.

10.2.4.2 Nitrogen Flow System

10.2.4.2.1 Turn on flow of Nitrogen to system. Check to assure the incoming pressure does not exceed 30 pounds per square inch (psi) on pressure gauge.

10.2.4.2.2 Adjust flow rate to 10 liters per minute (lpm) on flow rotometer.

10.2.4.2.3 Check to ensure all needles are in good working order and clean.

10.2.4.2.4 If any issues, notify team lead, supervisor, or manager.

10.3 **Pesticides by SW846 Method 8081, PCBs by SW846 Method 8082 (Microwave, Buchi Concentrator, & N-EVAP).**

10.3.1 Confirm all samples present and delivered, listed on the LIMS batch sheet, and listed on preparation log. If any discrepancies, check with schedulers, team leads, supervisors, or managers.

10.3.2 If no discrepancies, document custody of samples in Internal Chain program and change status of batch to "ASN" reflecting the initiation of extraction in LIMS.

10.3.3 Label all apparatuses with the correct sample identification. Sample labels may transfer from one apparatus to the next.

10.3.4 For all Pesticide and PCB samples follow the procedure below.

10.3.4.1 Weigh out two aliquots of minimum 15 grams to approximately 17 grams of Ottawa Sand into two separate tared, widemouth glass jars. One for a method blank and one for a blank spike.

10.3.4.2 Mix sample thoroughly and weigh out minimum 15 grams to approximately 17 grams of wet weight sample into a tared, widemouth jar. Record the weight to the nearest 0.1g, bottle number, if decanting performed, and sample description in the logbook.

Note: Samples which contain rocks, metal shavings, have a gasoline or oil odor, sludges, pure carbon (charcoal, activated carbon, etc), or low density (fabric, aerated soils, rubbers) may not be appropriate for this extraction. These may require a reduced sample amount, alternate extraction technique, and comments to be added. Pure Oils and Aqueous Samples are not appropriate for this extraction method. Check with the team leads, supervisors, or managers for further instructions.

10.3.4.3 Quality Control sample. For each batch, one sample will have two additional aliquots of minimum 15 grams to approximately 17 grams of wet weight sample weighed out into two separate tared, widemouth glass jars. One for Matrix Spike and one for Matrix Spike Duplicate.

10.3.5 Add drying agent to each sample (and including all QC samples) and mix to form a free flowing texture (sandy).

10.3.5.1 For baked hydromatrix, add up to 15g to each sample.

10.3.5.2 Sodium sulfate is NOT to be used as the drying agent for this prep method.

10.3.6 Transfer sample from widemouth jar to microwave tube.

10.3.7 Spiking & Surrogating. For proper execution protocols refer to EOP027.

10.3.7.1 Spiking Amounts. The test being requested will dictate the spike that will need to be added. Using a calibrated 1mL syringe, add spike to blank spike, matrix spike, and matrix spike duplicate (if present).

10.3.7.1.1 For Pesticide add 1mL of pesticide target compound spike.

10.3.7.1.2 For Pesticide Low Level (“LL”) add 0.1mL of pesticide target compound spike.

10.3.7.1.3 For PCB add 1mL of PCB target compound spike.

10.3.7.1.4 For PCB Low Level (“LL”) add 0.1mL of PCB target compound spike.

10.3.7.1.5 For samples testing for both Pesticide and PCB (known as “shared batch”), two separate sets of quality control samples must be prepared. One for Pesticides and one for PCBs.

Note: Check the expiration date for all spiking materials to verify that the solution is valid. Do not use expired solutions for spiking.

Note: Other special spikes maybe needed. Check the special spike section of the logbook for additional information.

10.3.7.2 Surrogate Amounts. The test being requested will dictate the amount of surrogate that will need to be added.

10.3.7.2.1 For Pesticide or PCB regular samples (not “LL”), add 1mL of the pesticide/PCB surrogate (labeled as “TCMX” on surrogate bottle) using a calibrated 1mL syringe to all samples, blanks, and spikes.

10.3.7.2.2 For Pesticide or PCB Low Level (“LL”) samples, add 0.1mL of the pesticide/PCB surrogate (labeled as “TCMX” on surrogate bottle) using a calibrated 1mL syringe to all samples, blanks, and spikes.

Note: Check the expiration date for all surrogating materials to verify that the solution is valid. Do not use expired solutions for surrogating.

Note: Other special surrogates maybe needed. Check the logbook for additional information.

10.3.8 Immediately add 30mL of 2:1 hexane:acetone solution to each sample, blanks, and spikes upon surrogate addition. Alternately, 20mL of hexane and 10mL of acetone can be added individually.

10.3.9 Attach the pressure cap (consists of two parts- Pressure Plug and Cap) and tighten securely using the tightening block.

10.3.10 Shake each capped extraction vessel to mix.

10.3.11 Microwave Extraction

10.3.11.1 Place all of the extraction vessels for the batch into the carousel. Balance out the carousel such that all samples are evenly distributed. Blank tubes (made by filling with approximately 15g Ottawa Sand and 30mL solvent) may be used to balance carousel.

10.3.11.2 Load the carousel into the Microwave extractor. Enter number of tubes (Sum of samples and blank tubes) in the carousel, select program “USEPA 3546-110”, and press “Start”. The microwave extractor will monitor the temperature of each vessel and adjust the power as needed. See Table 1 for recommended temperature program.

Note: If a sample explodes in the microwave, pause the program, wait at least 30 minutes before opening the microwave to allow the fumes to dissipate and the samples to cool down.

10.3.11.3 Once cycle is complete, open the microwave and remove the carousel. Unload the tubes from the carousel.

10.3.12 Transfer sample from microwave tube to Buchi tube.

10.3.12.1 Place a drying column on top of each Buchi tube.

10.3.12.2 Remove each cap and decant the liquid fraction through the baked sodium sulfate. Allow to filter through and collect in Buchi tube.

10.3.12.3 Using gentle tapping, add the solid sample to the filter and baked soduium sulfate.

10.3.12.4 Rinse each tube with approximately 2mL of hexane and pour through the appropriate funnel. Repeat 5 times.

10.3.12.5 Rinse the sample, baked sodium sulfate, and filter with 5-10mL of hexane and allow to drain through.

10.3.12.6 Immediately remove the filter funnel once dripping has stopped and swirl the extract. Samples are now ready for concentration.

10.3.13 Buchi Concentrator System

Note: In logbook, fill in Buchi concentrator information, chiller information, and flow rates in logbook once samples have been loaded.

10.3.13.1 Adjust hexane volumes to similar levels across all samples.

10.3.13.2 Load Buchi tubes into Buchi Concentrator. Use yellow plugs to close any Buchi tube slots that are not being used.

10.3.13.3 Select program “Pest/PCB Hex X”, and press “Start” on the vacuum controller. Turn rotation knob until digital display reads 250 rotations per minute. The extract is concentrated to less than 5ml.

10.3.13.4 Once program is finished, press “Stop” on vacuum controller and turn rotation knob to 0 rotations per minute.

10.3.13.5 Unload Buchi tubes from Buchi Concentrator and allow to cool.

10.3.14 Final volume adjustment. Samples may require further concentration or addition to reach proper volumes.

10.3.14.1 For Methods 8081, 8082, and “Shared Batch”, final volume is 10mL. Bring final volume to 10mL by adding additional Hexane to sample in class ‘A‘ Buchi tubes. Mix using the vortex mixer for 10 seconds minimum.

10.3.14.2 For 8081 Low Level (“8081 LL”) and 8082 Low Level (“8082 LL”), final volume is 1mL.

10.3.14.3 If samples will not concentrate to final volumes or change states (i.e. liquid to gel), notify team lead, supervisors, and managers for further instructions. These may require a modified sample amount, alternate concentration technique, and comments to be added.

10.3.14.4 N-EVAP Concentration Method. Immerse the nipple of the Buchi tube in the waterbath of the N-EVAP. Direct a gentle stream of nitrogen gas onto the extract by adjusting height of needle until small dimple appears on surface. Evaporate the solvent until the desired volume is reached. Occasionally rinse the side-walls of the concentrator tubes with hexane during the evaporation step.

10.3.15 Vialing

10.3.15.1 Sample Vial Labeling

10.3.15.1.1 For 8081 Low Level (“8081 LL”) and 8082 Low Level (“8082 LL”), sample, label one 2mL graduated amber vials with Sample number and Batch number. No backup vial is created for this test.

10.3.15.1.2 For 8081 and 8082 not including “Shared Batch” sample. Label two 2mL graduated amber vials with Sample number and Batch number. One for analysis and one for backup.

10.3.15.1.3 “Shared Batch” Low Level is not possible for soil samples. This is due to the cleanup procedure (EOP3665 Sulfuric Acid Cleanup and EOP3620 Florisil Cleanup) requiring sample volume of 1mL for each cleanup.

10.3.15.1.4 For “Shared Batch” sample. Label three 2mL graduated amber vials with Sample number and Batch number. One for PCB analysis, one for pesticide analysis and one for backup.

10.3.15.2 Vialing Backup. For 8081, 8082, and “Shared Batch”, transfer 2mL of extract into 2mL amber vial as a backup and do not add copper. Pesticide and PCB backup samples are vialed prior to appropriate cleanup.

10.3.15.3 Cleanups. Sample extract will require further cleanups prior to final vialing.

10.3.15.3.1 Sulfuric Acid Cleanup (For PCB analysis using SW8082)- Follow EOP3665.

10.3.15.3.2 Florisil Cleanup (For Pesticide analysis using SW8081)- Follow EOP3620.

10.3.15.3.3 For “Shared Batch”, a portion of the final volume of 10mL will be used for Sulfuric Acid Cleanup and a separate portion used for Florisil Cleanup.

10.3.15.4 Vialing Sample

Note: For vialing PCB sample destined for analysis, utilize the sulfuric acid cleanup extract. Final volume for 8082 and “Shared Batch” is 10mL. Final volume for 8082 LL is 1mL.

Note: For vialing Pesticide sample destined for analysis, utilize the florisil cleanup extract. Final volume for 8081 and “Shared Batch” is 10mL. Final volume for 8081 LL is 1mL.

10.3.15.4.1 For 8081, 8081 LL, 8082, and 8082 LL not including “Shared Batch” sample, transfer 1mL of extract into 2mL amber vial and copper cleanup following EOP011.

10.3.15.4.2 For “Shared Batch”, transfer two separate 1mL aliquots of extract into 2mL amber vials and copper cleanup both following EOP011. One for Pesticide and one for PCB.

10.3.15.5 Discard unvialed remainder of the extract into hexane wastes.

10.3.15.6 Protect the extracts from light and store them in the designated refrigerator at 4°C.

10.3.16 Upon completion of extraction and vialing, account for all samples by matching to logbook entry and confirm the following information is complete and present. This information must be noted in real-time and not after completion.

10.3.16.1 Method. Start date and time, stop date and time, and method used

10.3.16.2 Sample. Matrix type and weights

10.3.16.3 Extract. Colors, final volumes, and cleanup procedures

10.3.16.4 Reagent. Reagents used, lot numbers, and volumes used

10.3.16.5 Spikes and surrogates. Lot numbers, concentration, and volumes used

10.3.16.6 Equipment. Equipment used, flow rates, and temperatures.

10.3.16.7 Personnel. Document all personnel that have assisted in the extraction of the batch by having that person initial the logbook.

Note: It is not permissible under any circumstances to have another person initial for another person or take credit for work not performed.

10.3.17 In LIMS program, update status of samples to “DONE”, using associated batch number, date of extraction, department, select samples being updated using “X”, and saving changes. Document completion of sample extraction in Internal Chain program by changing status of batch to “OPEXT”.

10.3.18 Present samples and logbook to team leads, supervisors, or managers for final review.

10.3.19 Deliver samples for analysis. Protocols and expectations for delivery are dictated by analytical department and are expected to be adhered to.

10.4 **Acid-Base Neutrals by SW846 8270 (Microwave, Waterbath, & N-EVAP).**

10.4.1 Confirm all samples present and delivered, listed on the LIMS batch sheet, and listed on preparation log. If any discrepancies, check with schedulers, team leads, supervisors, or managers.

10.4.2 If no discrepancies, document custody of samples in Internal Chain program and change status of batch to “ASN” reflecting the initiation of extraction in LIMS.

10.4.3 Label all apparatuses with the correct sample identification. Sample labels may transfer from one apparatus to the next.

10.4.4 For all samples follow the procedure below.

10.4.4.1 Weigh out two aliquots of minimum 30g to approximately 32g of Ottawa Sand into two separate tared, widemouth glass jars. One for a method blank and one for a blank spike.

10.4.4.2 Mix sample thoroughly and weigh minimum 30g to approximately 32g of wet weight sample into a tared, widemouth jar. Record the weight to the nearest 0.1g, bottle number, if decanting performed, and sample description in the logbook.

Note: Samples which contain rocks, metal shavings, have a gasoline or oil odor, sludges, pure carbon (charcoal, activated carbon, etc), or low density (fabric, aerated soils, rubbers) may not be appropriate for this extraction These may require a reduced sample amount, alternate extraction technique, and comments to be added. Pure Oils and Aqueous Samples are not appropriate for this extraction method. Check with the team leads, supervisors, or managers for further instructions.

10.4.4.3 Quality Control sample. For each batch, one sample will have two additional aliquots of minimum 30g to approximately 32g of wet weight sample weighed out into two separate tared, widemouth glass jars. One for Matrix Spike and one for Matrix Spike Duplicate.

10.4.5 Add drying agent (baked hydromatrix) to each sample (and including all QC samples) and mix to form a free flowing texture (sandy).

10.4.5.1 For baked hydromatrix, add up to 15g to each sample.

10.4.5.2 Baked sodium sulfate is NOT to be used as the drying agent for this prep method.

10.4.6 Transfer sample from widemouth jar to microwave tube.

10.4.7 Spiking & Surrogating. For proper execution protocols refer to EOP027.

10.4.7.1 Spiking Amounts. The test being requested will dictate the spike that will need to be added. Using a calibrated 1mL syringe add spike to blank spike, matrix spike, and matrix spike duplicate (if present).

10.4.7.1.1 For Acid Base-Neutral ("ABN") add 1.0mL each of the ABN mix1 and ABN mix2.

10.4.7.1.2 For SIM, add 1.0mL of SIM spike.

Note: Check the expiration date for all spiking materials to verify that the solution is valid. Do not use expired solutions for spiking.

Note: Other special spikes maybe needed. Check the special spike section of the logbook for additional information.

10.4.7.2 Surrogate Amounts. The test being requested will dictate the surrogate that will need to be added.

10.4.7.2.1 For "ABN" or "SIM" samples, add 1.0mL of the ABN surrogate using a calibrated 1mL syringe to all samples, blanks, and spikes.

10.4.7.2.2 For "ABN DOD SIM" samples, add 1.0mL of the "ABN DOD SIM" surrogate using a calibrated 1mL syringe to all samples, blanks, and spikes.

Note: Check the expiration date for all surrogating materials to verify that the solution is valid. Do not use expired solutions for surrogating.

Note: Other special surrogates maybe needed. Check the logbook for additional information.

10.4.8 Immediately add 30mL of 1:1 methylene chloride:acetone solution to each sample, blanks, and spikes upon surrogate addition. Alternately, 15mL of methylene chloride and 15mL of acetone can be added individually.

10.4.9 Attach the pressure cap (consists of two parts- Pressure Plug and Cap) and tighten securely using the tightening block.

10.4.10 Shake each capped extraction vessel to mix.

10.4.11 Microwave Extraction

10.4.11.1 Place all of the extraction vessels for the batch into the carousel. Balance out the carousel such that all samples are evenly distributed. Blank tubes (made by filling with approximately 15g Ottawa Sand and 30mL solvent) may be used to balance carousel.

10.4.11.2 Load the carousel into the Microwave extractor. Enter number of tubes (Sum of samples and blank tubes) in the carousel, select program “USEPA 3546-110”, and press “Start”. The microwave extractor will monitor the temperature of each vessel and adjust the power as needed. See Table 1 for recommended temperature program.

Note: If a sample explodes in the microwave, pause the program, wait at least 30 minutes before opening the microwave to allow the fumes to dissipate and the samples to cool down.

10.4.11.3 Once cycle is complete, open the microwave and remove the carousel. Unload the tubes from the carousel.

10.4.12 Transfer sample from microwave tube to Kuderna-Danish.

10.4.12.1 Place a drying column on the top of the assembled Kuderna-Danish.

10.4.12.2 Remove each cap and decant the liquid fraction through the drying column. Allow to filter through and collect in Kuderna-Danish.

10.4.12.3 Using gentle tapping, add the solid sample to the drying column.

10.4.12.4 Rinse each tube with approximately 2mL of methylene chloride and pour through the appropriate funnel. Repeat up to 5 times.

10.4.12.5 Rinse the sample, baked sodium sulfate, and filter with 5-10mL of methylene chloride and allow to drain through.

10.4.12.6 Immediately remove the filter funnel once dripping has stopped and discard sodium sulfate, glass wool or filter paper into solid sulfate waste.

10.4.12.7 Add in boiling chip and Snyder column. Samples are now ready for concentration.

10.4.13 Sample Concentration Procedure. Document the procedure in the extraction logbook.

10.4.13.1 Water Bath with Evaporation Collection System

Note: Fill in waterbath information, chiller information, and flow rates once samples have been loaded.

Note: If a sample ruptures into the waterbath, immediately close the hood, allow the boiling action of the water to subside, fumes to dissipate, and notify a team lead, supervisor, or manager. Do not attempt glassware removal or cleanup by yourself.

10.4.13.1.1 Position K-D flask in the waterbath. Immersing the 10mL concentrator tube into the waterbath. Concentrate samples to about 5-7mL.

10.4.13.1.2 Allow the K-D flask, receiver, and Snyder column to cool. During this process, approximately 1mL of methylene chloride will recondense and flow down the side of the K-D flask removing most of the extract remaining in the flask and joints.

10.4.13.1.3 Remove Snyder column and slowly add 0.5-1mL of methylene chloride to the tilted K-D flask while rotate the flask. This is a final rinse of the flask and joint. Tilt upright to allow the methylene chloride to drain into the receiver.

10.4.13.1.4 Disassemble the K-D flask from the receiver.

10.4.14 Final volume adjustment. Samples may require further concentration or addition to reach proper volumes.

10.4.14.1 For Methods 8270 and SIM, final volume is 1mL. If samples will not concentrate to final volumes or change states (i.e. liquid to gel), notify team lead, supervisors, and managers for further instructions. These may require a modified sample amount, alternate concentration technique, and comments to be added.

10.4.14.2 N-EVAP Concentration Method. Immerse the nipple of the receiver in the waterbath of the N-EVAP. Direct a gentle stream of nitrogen gas onto the extract by adjusting height of needle until small dimple appears on surface. Evaporate the solvent until the desired volume is reached. Occasionally rinse the side-walls of the concentrator tubes with methylene chloride during the evaporation step.

10.4.15 Vialing

10.4.15.1 For each sample, label a 2mL graduated clear vial with Sample number and Batch number. No backup vial is created for this test.

10.4.15.2 Bring final volume to 1mL with methylene chloride in receiver and mix with a Pasteur pipet. Transfer all sample extract to the vial.

10.4.15.3 Adjust the vial volume to 1mL with methylene chloride.

10.4.15.4 Store the extracts in the designated freezer at -10°C.

10.4.16 Upon completion of extraction and vialing, account for all samples by matching to logbook entry and confirm the following information is complete and present. This information must be noted in real-time and not after completion.

10.4.16.1 Method. Start date and time, stop date and time, and method used

10.4.16.2 Sample. Matrix type and weights

10.4.16.3 Extract. Colors, final volumes, and cleanup procedures

10.4.16.4 Reagent. Reagents used, lot numbers, and volumes used

10.4.16.5 Spikes and surrogates. Lot numbers, concentration, and volumes used

10.4.16.6 Equipment. Equipment used, flow rates, and temperatures.

10.4.16.7 Personnel. Document all personnel that have assisted in the extraction of the batch by having that person initial the logbook.

Note: It is not permissible under any circumstances to have another person initial for another person or take credit for work not performed.

10.4.17 In LIMS program, update status of samples to "DONE", using associated batch number, date of extraction, department, select samples being updated using "X", and saving changes. Document completion of sample extraction in Internal Chain program by changing status of batch to "OPEXT".

10.4.18 Present samples and logbook to team leads, supervisors, or managers for final review.

10.4.19 Deliver samples for analysis. Protocols and expectations for delivery are dictated by analytical department and are expected to be adhered to.

10.5 **DRO - Diesel Range Organics for method SW846-8015 (Microwave, Buchi Concentrator/Waterbath, & N-EVAP).**

10.5.1 Confirm all samples present and delivered, listed on the LIMS batch sheet, and listed on preparation log. If any discrepancies, check with schedulers, team leads, supervisors, or managers.

10.5.2 If no discrepancies, document custody of samples in Internal Chain program and change status of batch to "ASN" reflecting the initiation of extraction in LIMS.

10.5.3 Label all apparatuses with the correct sample identification. Sample labels may transfer from one apparatus to the next.

10.5.4 For all DRO samples except Fingerprint ("FING") follow the procedure below.

10.5.4.1 Weigh out two aliquots of minimum 10g to approximately 12g of Ottawa Sand into two separate tared, widemouth glass jars. One for a method blank and one for a blank spike.

10.5.4.2 Mix sample thoroughly and weigh out minimum 10g to approximately 12g of wet weight sample into a tared, widemouth jar. Record the weight to the nearest 0.1g, bottle number, if decanting performed, and sample description in the logbook.

Note: Samples which contain rocks, metal shavings, have a gasoline or oil odor, sludges, pure carbon (charcoal, activated carbon, etc), or low density (fabric, aerated soils, rubbers) may not be appropriate for this extraction. These may require a reduced sample amount, alternate extraction technique, and comments to be added. Pure Oils and Aqueous Samples are not appropriate for this extraction method. Check with the team leads, supervisors, or managers for further instructions.

10.5.4.3 Quality Control sample. For each batch, one sample will have two additional aliquots of minimum 10g to approximately 12g of wet weight sample weighed out into two separate tared, widemouth glass jars. One for Matrix Spike and one for Matrix Spike Duplicate.

10.5.5 For DRO Fingerprint ("FING") follow the procedure below.

10.5.5.1 Weigh out one aliquot of minimum 10g to approximately 12g of Ottawa Sand into a separate tared, widemouth glass jar for a method blank.

10.5.5.2 Mix sample thoroughly and weigh out minimum 10g to approximately 12g of wet weight sample into a tared, widemouth jar. Record the weight to the nearest 0.1g, bottle number, if decanting performed, and sample description in the logbook.

Note: Samples which contain rocks, metal shavings, have a gasoline or oil odor, sludges, pure carbon (charcoal, activated carbon, etc), or low density (fabric, aerated soils, rubbers) may not be appropriate for this extraction. These may require a reduced sample amount or alternate extraction technique. Pure Oils and Aqueous Samples are not appropriate for this extraction method. Check with the team leads, supervisors, or managers for further instructions.

10.5.5.3 Quality Control sample. Fingerprint does not have any quality control samples.

10.5.6 Add drying agent (baked hydromatrix) to each sample (and including all QC samples) and mix to form a free flowing texture (sandy).

10.5.6.1 For baked hydromatrix, add up to 10g to each sample.

10.5.6.2 Baked sodium sulfate is NOT to be used as the drying agent for this prep method.

10.5.7 Transfer sample from widemouth jar to microwave tube.

10.5.8 Spiking & Surrogating. For proper execution protocols refer to EOP027.

10.5.8.1 Spiking Amounts. The test being requested will dictate the spike that will need to be added. Using a calibrated 1mL syringe, add spike to blank spike, matrix spike, and matrix spike duplicate (if present).

10.5.8.1.1 Add 1mL of the DRO target compound spike to the samples selected for matrix spikes.

Note: Check the expiration date for all spiking materials to verify that the solution is valid. Do not use expired solutions for spiking.

10.5.8.2 Surrogate Amounts. Add 1mL of the DRO surrogate using a calibrated 1mL syringe to all samples, blanks, and spikes.

10.5.8.3 For DRO Fingerprint (FING), add 4ml of DRO surrogate using a calibrated syringe to all samples and method blank

Note: Check the expiration date for all surrogating materials to verify that the solution is valid. Do not use expired solutions for surrogating.

10.5.9 Immediately add 30mL of 1:1 methylene chloride:acetone solution to each sample, blanks, and spikes upon surrogate addition. Alternately, 15mL of methylene chloride and 15mL of acetone can be added individually.

10.5.10 Attach the pressure cap (consists of two parts- Pressure Plug and Cap) and tighten securely using the tightening block.

10.5.11 Shake each capped extraction vessel to mix.

10.5.12 Microwave Extraction

10.5.12.1 Place all of the extraction vessels for the batch into the carousel. Balance out the carousel such that all samples are evenly distributed. Blank tubes (made by filling with approximately 15g Ottawa Sand and 30mL solvent) may be used to balance carousel.

10.5.12.2 Load the carousel into the Microwave extractor. Enter number of tubes (Sum of samples and blank tubes) in the carousel, select program “USEPA 3546-110”, and press “Start”. The microwave extractor will monitor the temperature of each vessel and adjust the power as needed. See Table 1 for recommended temperature program.

Note: If a sample explodes in the microwave, pause the program, wait at least 30 minutes before opening the microwave to allow the fumes to dissipate and the samples to cool down.

10.5.12.3 Once cycle is complete, open the microwave and remove the carousel. Unload the tubes from the carousel.

10.5.13 Transfer sample from microwave tube to Buchi tube.

10.5.13.1 Place a filter paper and baked sodium sulfate on the top of each Buchi tube.

10.5.13.2 Remove each cap and decant the liquid fraction through the baked sodium sulfate. Allow to filter through and collect in Buchi tube.

10.5.13.3 Using gentle tapping, add the solid sample to the filter and baked sodium sulfate.

10.5.13.4 Rinse each tube with approximately 2mL of methylene chloride and pour through the appropriate funnel. Repeat up to 5 times.

10.5.13.5 Rinse the sample, baked sodium sulfate, and filter with 5-10mL of methylene chloride and allow to drain through.

10.5.13.6 Immediately remove the filter funnel once dripping has stopped and swirl the extract. Samples are now ready for concentration.

10.5.14 Buchi Concentrator System (Waterbath is also permissible for methylene chloride concentration)

10.5.14.1 Group samples into groups of 12. Use yellow plugs to close any Buchi tube slots that are not being used.

10.5.14.2 Adjust volumes to similar levels across all samples using methylene chloride.

10.5.14.3 Load Buchi tubes into Buchi Concentrator.

10.5.14.4 Select program “DRO/EPHNF”, and press “Start” on the vacuum controller. Turn rotation knob until digital display reads 250 rotations per minute. The extract is concentrated to approximately 1mL.

10.5.14.5 Once program is finished, press “Stop” on vacuum controller and turn rotation knob to 0 rotations per minute.

10.5.14.6 Unload Buchi tubes from Buchi Concentrator and allow to cool.

10.5.15 Final volume adjustment. Samples may require further concentration or addition to reach proper volumes.

10.5.15.1 For Method 8015 and “FING", final volume is 1mL. If samples will not concentrate to final volumes or change states (i.e. liquid to gel), notify team lead, supervisors, and managers for further instructions. These may require a modified sample amount, alternate concentration technique, and comments to be added.

10.5.15.2 N-EVAP Concentration Method. Immerse the nipple of the Buchi tube in the waterbath of the N-EVAP. Direct a gentle stream of nitrogen gas onto the extract by adjusting height of needle until small dimple appears on surface. Evaporate the solvent until the desired volume is reached. Occasionally rinse the side-walls of the concentrator tubes with methylene chloride during the evaporation step.

10.5.16 Vialing

10.5.16.1 For each sample, label a 2mL graduated amber vial with Sample number and Batch number. No backup vial is created for this test.

10.5.16.2 Bring final volume to 1mL with methylene chloride in receiver and mix with a Pasteur pipet. Transfer all sample extract to the vial.

10.5.16.3 Adjust the vial volume to 1mL with methylene chloride.

10.5.16.4 Protect the extracts from light and store them in the designated refrigerator at 4°C.

10.5.17 Upon completion of extraction and vialing, account for all samples by matching to logbook entry and confirm the following information is complete and present. This information must be noted in real-time and not after completion.

10.5.17.1 Method. Start date and time, stop date and time, and method used

10.5.17.2 Sample. Matrix type and weights

10.5.17.3 Extract. Colors, final volumes, and cleanup procedures

10.5.17.4 Reagent. Reagents used, lot numbers, and volumes used

10.5.17.5 Spikes and surrogates. Lot numbers, concentration, and volumes used

10.5.17.6 Equipment. Equipment used, flow rates, and temperatures.

10.5.17.7 Personnel. Document all personnel that have assisted in the extraction of the batch by having that person initial the logbook.

Note: It is not permissible under any circumstances to have another person initial for another person or take credit for work not performed.

10.5.18 In LIMS program, update status of samples to “DONE”, using associated batch number, date of extraction, department, select samples being updated using “X”, and saving changes. Document completion of sample extraction in Internal Chain program by changing status of batch to “OPEXT”.

10.5.19 Present samples and logbook to team leads, supervisors, or managers for final review.

10.5.20 Deliver samples for analysis. Protocols and expectations for delivery are dictated by analytical department and are expected to be adhered to.

11.0 CALCULATIONS

11.1 Not applicable.

12.0 QUALITY ASSURANCE

12.1 Extract a method blank and blank spike at a rate of one per day or every twenty (20) samples, whichever is more frequent.

12.2 A matrix spike / matrix spike duplicate (MS/MSD) is required per every 20 samples.

12.3 For pest/PCBs a separate BSP and MS/MSD set are needed if the sample requires PCBs, Toxaphene and/or chlordane. Therefore, if a sample is to be extracted for pesticides and PCBs, 2 sets of BS, MS, and MSD are needed.

12.4 Refer to Project Specific Bench Notes (GC8081, GC8082, MS8270) for additional program or client specific QC requirements.

12.5 Perform solvent checks for each new solvent lot to verify the absence of interferences. See SOP EOP013.

13.0 DOCUMENTATION

13.1 All sample preparation activities and related information must be documented in the respective extraction logbook. Complete all information required for the extraction summary logbook.

13.2 Errors must be stricken with a single line, initialed and dated. The correct information must be written adjacent to the erroneous information.

13.3 The individual with custody responsibility must sign the extraction log.

13.4 The individuals involved in the processing of the batch must initial in the appropriate respective locations.

13.5 All spikes, surrogates, reagents, solutions, and applicable manufacturer and lot information must be filled in the appropriate respective locations.

13.6 All equipment, equipment identifiers, and the operating parameters at time of use must be filled in the appropriate respective locations.

13.7 The logbooks and/or e-logs must be reviewed and initialed by a team leader or supervisor. The approved page is copied and given to the Report Generation Dept.

13.8 Sample custody transfers must be updated according to proper Chain of Custody protocols.

13.9 All standards preparation must be documented in the standards preparation logbook.

13.10 Equipment maintenance logs must be maintained.

14.0 DATA REPORTING

14.1 Not Applicable.

15.0 POLLUTION PREVENTATION & WASTE MANAGEMENT

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.3 Non-hazardous aqueous waste

15.4 Hazardous aqueous wastes

15.5 Chlorinated organic solvents

15.6 Non-chlorinated organic solvents

15.7 Hazardous solid wastes

15.8 Non-hazardous solid waste

16.0 REFERENCES

16.1 No additional references are required.

Table 1

Recommended Temperature Programs for Microwave Ovens

Mars-express Microwave:

Program - USEPA 3546-110 is used for Acid/Base Neutrals, Pesticides/PCBs, DRO, NJEPH, and Herbicides

Ramp to 110°C in 15 minutes
Hold at 110°C for an additional 15 minutes
Cool for 15 minutes

Mar 5 Microwave:

Program - USEPA 3546-110 is used for Acid/Base Neutrals, Pesticides/PCBs, DRO, NJEPH, and Herbicides

Ramp to 110°C in 15 minutes
Hold at 110°C for an additional 15 minutes
Cool for 15 minutes

Mar 6 Microwave:

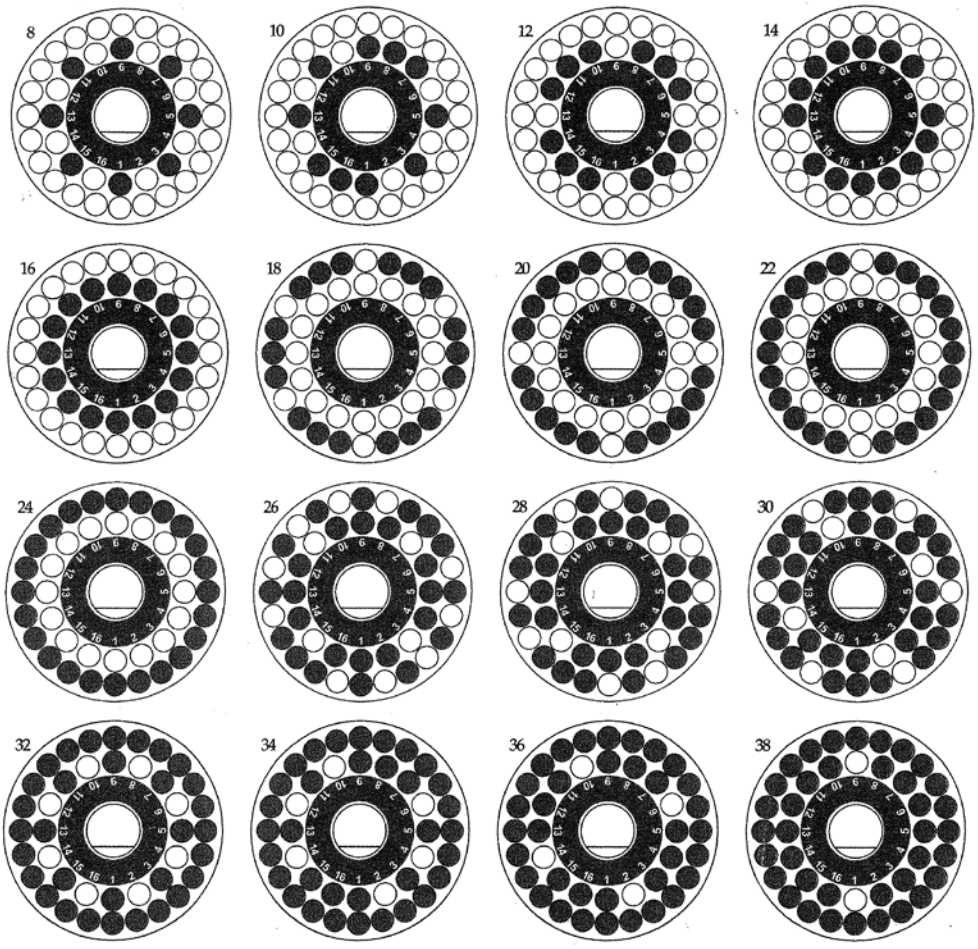
Program - USEPA 3546-110 is used for Acid/Base Neutrals, Pesticides/PCBs, DRO, NJEPH, and Herbicides

Ramp to 110°C in 15 minutes
Hold at 110°C for an additional 15 minutes
Cool for 15 minutes

Table 2

(from Mars User Guidelines Book 600288 - Rev.1)

Recommended distribution of MARSXpress vessels when turntable is less than capacity



Incorrect distribution: What not to do

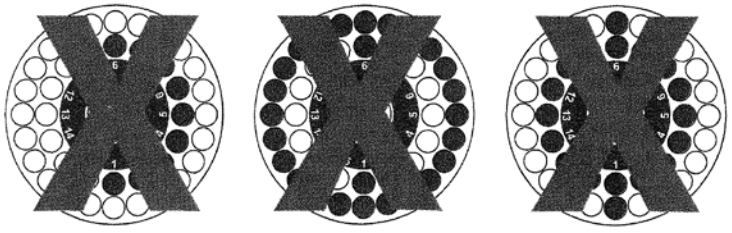


Table 3

Recommended Programs for Buchi Concentrator System

Buchi Program Name	Step	Pressures (in mbar)		Time (in minutes)
		Start	End	
DRO/EPHNF	1	1000	450	1
	2	450	450	9
	3	375	375	7
	4	275	275	4
	5	200	200	2
	6	150	150	4
8081/8082 3546	1	1000	450	1
	2	425	425	6
	3	375	375	5
	4	275	275	1
P/P Hex X	1	1000	500	2
	2	475	475	3
	3	400	400	5
	4	300	300	10
	5	200	200	5
	6	125	125	5
EPH (Pt I)/PPCB LVI	1	1000	450	1
	2	450	450	9
	3	375	375	5
EPH (Pt II)	1	1000	500	2
	2	475	475	3
	3	400	400	5
	4	300	300	10
	5	200	200	5
	6	125	125	5
ARO	1	1000	900	3
	2	900	800	4
	3	800	700	2
	4	700	600	5
	5	600	500	1

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
10.3.12.4	Clarified how many time to rinse with 2 ml of solvent

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
25	09/07/2021	Angela Lattanzio
26	9/20/2022	Olga Azarian

END OF DOCUMENT

LAB SUPERVISOR: Bridget Kelly

QA OFFICER: Maree Kuschke

EFFECTIVE DATE: 11-30-2023

TITLE: PURGE-AND-TRAP EXTRACTION FOR AQUEOUS SAMPLES
REFERENCES: SW846 8260D (REVISION 3, AUGUST 2006), SW846 8000D(REVISION 4, JULY 2014), SW846 5030C
REVISED SECTIONS: *UPDATED METHOD REVISIONS; 6.1, 7.5.3, 10.2.5.1.1, 10.2.3, 10.2.7.3.3, 10.3.2.2*

DELETE SCTIONS:6.2

1.0 SCOPE AND APPLICATION

1.1 This method describes a purge-and-trap procedure for the analysis of volatile organic compounds (VOCs) in aqueous samples, water miscible liquid, and high-level soil and waste sample extracts prepared in Method 5035A. This method is used in conjunction with gas chromatographic determinative methods SW8015 and SW 8260.

1.2 Method 5030C can be used for most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

1.3 Water samples can be analyzed directly for volatile organic compounds by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in water can be determined by direct injection of the sample into the chromatographic system or by dilution of the sample prior to the purge-and-trap process.

2.0 SUMMARY

2.1 Aqueous Samples: An inert gas is bubbled through a portion of the aqueous sample at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column.

2.2 High Concentration Extracts from Method 5035A: An aliquot of the extract prepared in Method 5035A is combined with organic free reagent water in the purging chamber. It is then analyzed by purge-and-trap GC or GC/MS following the normal aqueous method.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT – N/A

4.0 DEFINITIONS

BATCH - a group of samples which are similar with respect to matrix and testing procedures being employed and which are processed as a unit. A sample batch is a maximum of 20 samples which can be prepared and analyzed over a period of one day.

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank; Instrument Blank, Storage Blank, and Sulfur Blank.

MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards, that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

REAGENT WATER - water in which an interferant is not observed at or above the minimum detection limit of the parameters of interest.

TRIP BLANK - a sample of analyte free matrix taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor/Health and Safety Officer.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 Primary standards of the toxic compounds must be prepared in a hood. A NIOSH/Mass approved toxic gas respirator must be worn when the analyst handles high concentrations of these toxic compounds.

6.0 COLLECTION, PRESERVATION, & HOLDING TIMES

6.1 Collect samples in 40 ml glass screw-cap VOA vials with Teflon-faced silicone septum.

6.2 Preserve samples with HCl to pH ≤2. Samples received unpreserved must be so noted on the chain of custody. Project Manager are notified of the non-conformance, the client is contacted. The lab waits for additional instructions The non-conformance is documented in the report narrative.

6.3 Store samples with minimum headspace, at 4 °C or less in an area free of solvent fumes. The size of any bubble caused by degassing upon cooling the sample must not exceed 5 - 6 mm. When a bubble is present, also observe the cap and septum to ensure that a proper seal was made at time of sampling. If the sample was improperly sealed, the sample must be discarded.

6.4 Samples must be analyzed within 14 days of collection.

7.0 APPARATUS & MATERIALS

7.1 Microsyringes - 10-µL, 25-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL.

7.2 Syringe valve - Two-way, with Luer ends.

7.3 5-mL glass hypodermic syringes with Luer-Lok tip.

7.4 Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers.

7.5 Purge-and-trap device.

7.5.1 The following autosampler models are used for purging, trapping, and desorbing the sample into GC column.

- O.I. Model 4560 sample concentrator with 4551 vial multi-sampler.
- O.I. Model 4560 sample concentrator with 4552 Water/Soil multi-sampler.

7.5.2 The sample purge chamber accepts 5 ml samples with a water column at least 3 cm deep.

7.5.3 The EST auto sampler is equipped with a heater capable of maintaining the purge chamber at 40 °C (± 1°C) is to be used for low level soil/sediment analysis, but not for water or medium level soil/sediment analyses.

7.5.4 The trap is 25 cm long with an inside diameter of 0.105 inch, which is purchased from a commercial vendor. It contains Tenax, silica gel, and a carbon molecular sieve. Before initial use, the trap must be conditioned at 180°C for 30 minutes by backflushing with at least 20 ml/minute flow of helium.

7.5.4.1 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analyte of interest. Whichever trap is employed, it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in this method.

7.5.5 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap must not be heated higher than 180 °C and the remaining sections must not exceed 210 °C during bakeout mode.

7.6 Vials - 40 ml with Teflon lined septa.

7.7 pH paper

8.0 STANDARDS & REAGENTS

8.1 Reagent Water

8.1.1 Reagent water is defined as water in which an interferant is not observed at the method detection limit of the parameters of interest.

8.1.2 Reagent water is generated by using a multi-element deionizing system consisting of a particulate filter, twin mixed bed ion exchange resin columns, and a carbon-polishing column.

8.2 Methanol - purge-and-trap grade or equivalent.

8.3 Refer to the determinative method SOP for specifications on internal and surrogate standards.

9.0 INTERFERENCES

9.1 The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

9.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

9.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it must be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to cross contamination from highly concentrated samples. Frequently bake-out and purge the entire system on a routine basis.

9.4 Special precautions must be taken to control methylene chloride sources. All GC carrier gas lines and purge gas plumbing must be constructed of stainless steel or copper tubing. Organics prep staff whose clothing is routinely exposed to methylene chloride and other solvent vapors during sample preparation are banned from wearing their lab coat in the GC/MS laboratory.

10.0 PROCEDURE

10.1 The purge-and-trap technique for aqueous samples is found in Sec. 10.2 and guidance for analysis of solvent extracts from the High Concentration Method in Method 5035C is found in Sec. 10.3. The samples prepared by this method may be analyzed by Methods 8015 and 8260. Refer to these methods for appropriate analysis conditions.

10.2 This section provides guidance on the analysis of aqueous samples and samples that are water miscible, by purge-and-trap analysis.

10.2.1 Condition the trap according to manufacturer's instructions upon installation. Routine Daily maintenance must be performed before any tuning, calibration or sample analysis activities are initiated. These include checks of the following items:

- Clean & bake purge tube.
- Bake trap and transfer lines.
- Check or refill internal/surrogate spike solution on SIM/SAM vials.
- Clean/replace syringe (if necessary).
- Change and refill rinse bottle.
- Empty and rinse waste bottle.

10.2.2 Prior to using this introduction technique, the GC system must be calibrated. The procedures for the determinative methods give details on preparation of standards for initial and daily calibrations. The GC/MS methods require instrument tuning prior to proceeding with calibration.

10.2.2.1 Transfer and fill up (no air space) each standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray.

10.2.2.2 Program the autosampler to add surrogate and internal standard spiking solution to each standard.

10.2.2.3 Follow the purge-and-trap analysis as outlined in Sec. 10.2.4.

10.2.2.4 Calculate response factors (RF) or calibration factors (CF) for each analyte of interest. Follow the specific calibration requirements as described in the determinative method SOP.

10.2.3 Sample Screening

10.2.3.1 Screening of the sample prior to purge-and-trap analysis may provide guidance on whether sample dilution is necessary and may prevent contamination of the purge-and-trap system.

10.2.3.2 The samples are screened by GC orGC/MS .

10.2.4 Sample Introduction and Purging

10.2.4.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

10.2.4.2 Adjust the purge gas flow rate to 25-40 mL/min on the purge-and-trap device. Once the flow is optimized, it is not necessary to set the flow daily, although periodic checking is recommended.

10.2.4.3 Using EST,

- Place the 40 ml vial in the tray, or
- Load 5ml sample into purge tube if sample volume limited.

10.2.4.4 Program the autosampler to inject the internal standard (if applicable) and surrogate solution into the robotic syringe used to withdraw sample from the 40 ml vial.

10.2.4.5 Purge the sample for the time and at the temperature specified in the determinative method SOP.

10.2.5 Sample Desorption

10.2.5.1 Place the purge-and-trap system in the desorb mode, and rapidly heat the trap to the temperature specified in the determinative method SOP while backflushing with helium for the method specific time. Simultaneously begin the temperature program of the gas chromatograph and start the data acquisition.

10.2.5.1.1 Desorb time is 4 minutes for EPA 524.2 and 624. Desorb time for SW846 8260D can be 0.5 to 4 minutes.

10.2.5.2 Program the purge and trap system to automatically rinse purge tube at least twice with heated reagent water between analyses to avoid carryover of target compounds. For samples containing large amounts of water-soluble materials, suspended solids, high-boiling compounds, or high purgeable levels, it may be necessary to wash out the purging device with methanol solution between analyses, rinse it with distilled water.

10.2.6 Trap Reconditioning

10.2.6.1 Recondition the trap by returning the purge-and-trap device to the purge mode. Maintain the trap temperature and bake time as specified in the method SOP.

10.2.7 Sample Dilution

10.2.7.1 If the concentration of any target compound in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and reanalyzed.

10.2.7.2 Establish the dilution of sample in order to fall within the calibration range:

- Use screen data.
- Employ data from undiluted sample analysis.
- Use historical site data.
- Use sample characteristics (i.e. appearance, odor) as initial guidance.

10.2.7.3 The following procedure is appropriate for diluting purgeable samples. Until the dilute sample is in a sealed sample vial, all steps in the dilution procedure must be performed without delay.

10.2.7.3.1 Dilutions may be made in volumetric flasks (10-mL to 100- mL). Intermediate dilutions may be necessary for extremely large dilutions.

10.2.7.3.2 Calculate the approximate volume of reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask.

10.2.7.3.3 Inject the proper aliquot of samples from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask and invert.

10.2.7.3.4 Fill a 40 ml sample vial and seal with a Teflon baked silicon septa, load the diluted sample into the autosampler and analyze.

10.2.8 pH Verification.

10.2.8.1 Once the sample analysis is completed or before preparing sample dilution, the pH of the sample must be determined to ensure that all VOA samples were properly preserved in the field. Put one or two drops of sample directly on a piece of pH paper. Check the pH. Any sample with a pH greater than 2 will be recorded accordingly on the instrument run log and will be reported with a footnote indicating the sample preservation deficiency.

10.2.9 Analysis of water-miscible liquids

10.2.9.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with organic-free reagent water.

10.3 This section provides guidance on the analysis of solvent extracts from High Concentration Samples prepared by Method 5035A.

10.3.1 Low-level soil method.

10.3.1.1 Weigh out 5 g of each sample into a labeled vial. Add 5 ml of reagent water and cap the vial quickly. Transfer the 40 ml vial to the autosampler tray. Stir and heat the sample at the time of analysis.

10.3.2 Medium-level soil method.

10.3.2.1 Select the volume of methanol extract to add to the 50 ml of reagent water for analysis. If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-concentration analysis to determine the appropriate volume.

10.3.2.2 Aliquot 5 or 10 ml of solution into the 40 ml vial and cap with Teflon septum, then place the vial onto the autosampler.

10.3.3 Proceed with the analysis as outlined in the specific determinative method. Analyze all reagent blanks on the same instrument as that used for the samples. The standards and blanks must also contain the same amount of methanol to simulate the sample conditions.

11.0 QUALITY CONTROL

11.1 Analyze a method blank and blank spike at a rate of one per day or every twenty samples, whichever is more frequent.

11.2 A matrix spike/matrix spike duplicate is required for every 20 samples. Client specific requirements may specify a MS/MSD per day.

12.0 DOCUMENTATION

12.1 The Analytical Logbook records the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

12.1.1 If samples require reanalysis, a brief explanation of the reason must be documented in the Comments section.

12.2 Standards Preparation Logbook must be completed for all standard preparations. All information must be completed; the page must be signed and dated by the appropriate person.
12.2.1 The SGS lot number must be cross-referenced on the standard vial.

12.3 Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

12.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

12.5 Unused blocks of any form must be X'ed and Z'ed by the analyst before submitting the data for review.

12.6 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information must also be verified during this review.

13.0 POLLUTION PREVENTION & WASTE MANAGEMENT

13.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 13.2.

13.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, ESM003. This

document describes the proper disposal of all waste materials generated during the testing of samples as follows:

13.2.1 Non hazardous aqueous wastes.
13.2.2 Hazardous aqueous wastes
13.2.3 Chlorinated organic solvents
13.2.4 Non-chlorinated organic solvents
13.2.5 Hazardous solid wastes
13.2.6 Non-hazardous solid wastes

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
	SOP was revised from the the 5030 version.
6.1	Vials are purchased through a Vendor, not washed in house.
7.5.3	EST equipment replaced all other Models
10.2.5.1.1	Desorb for 524.2 & 624.1 is 4 minutes. SW8260D can be 0.5 – 4 minutes.
10.2.7.3.3	Removed – shake“
10.2.3	Samples are screened by GC or GCMS not SW846- 3810.

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed
6.2	Chlorine not tested, only pH.

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added

History of Revisions

Version #	Date of Revision	Revised By
01	11/27/2023	Maria Ruschke

END OF DOCUMENT

LAB SUPERVISOR: Bridget Nally

QA OFFICER: Maree Kerschke

EFFECTIVE DATE: 11-26-2023

TITLE: COLLECTION, PRESERVATION, MANAGEMENT AND HANDLING OF SOLID SAMPLES FOR VOLATILE ORGANICS ANALYSIS BY SW846 METHODOLOGY
REFERENCES: SW846, Method 5035A (Third Edition, Third Update, June 1997)
REVISED SECTIONS: Format, 1.3, 9.1.3.1, 9.2.2.2, 10.2.2.1, 11.4.1.4
ADDED SECTIONS: 8.1.1.3, 9.2.2.3

1.0 SCOPE AND APPLICATION

1.1 The following method describes the procedures used by SGS North America Inc - Dayton for the collection, preservation, management and handling of solid samples for volatile organics analysis using SW846 methodology.

1.2 This procedure is designed for the sampling and preservation of sediments, soils, filter cakes, oily wastes and tars,

1.3 This method is used in conjunction with other SW846 8000 series instrumental methods for an analytical finish (determinative method). This SOP describes the procedures employed for the sampling and sample preservation components of 5035A. Sample introduction techniques are described in the SOP for Methods SW846 8015D and SW846 8260D.

1.4 Techniques are described for collecting solid samples for analysis at two different target concentration ranges; low level concentration, <200 ug/kg and medium level concentration, >200ug/kg.

1.5 This procedure also describes specific steps for the handling of solid samples collected in Encore™ samplers.

2.0 SUMMARY OF METHOD

2.1 Solid samples are collected in the field using techniques that are designed to minimize volatilization loses incurred during sampling, transportation and storage prior to analysis. Samples are collected using a modified syringe or Encore™ sampler designed to hold approximately 5 grams of material.

2.2 Samples for low concentration analysis, <200 ug/kg, collected in the field in modified syringes are transferred to pre-weighed vials containing 5 ml of DI water. Low concentration samples collected in Encore™ samplers are transported to the laboratory and transferred to pre-weighed vials containing 5 ml of DI water at the laboratory.

2.3 Samples for medium concentration analysis, >200 ug/kg, collected in the field in modified syringes are transferred to pre-weighed vials containing 10 ml of methanol. Medium concentration samples collected in Encore™ samplers are transported to the laboratory and transferred to pre-weighed vials containing 5 ml of methanol.

2.4 Solid samples collected in the field using Encore™ samplers are logged in by the sample management staff and transferred to designated refrigerated storage. Information regarding the samples arrival and disposition are rapidly communicated to the organics staff. The organics staff transfers the sample from the Encore™ sampler into the designated preservative solution within the required time period, documents the transfer and places the preserved sample into storage for analysis.

2.5 Samples for low concentration analysis, <200 ug/kg, are transferred into Dl water and weighed. Samples for medium concentration, >200 ug/kg, are transferred to a methanol solution and weighed. Low level samples may also be transferred into a sodium bisulfate solution, at client request.

2.6 Three replicates of samples of unknown concentration are collected and transferred to both Dl water and methanol for use as dictated by the concentration of target analytes in the sample.

2.7 All transfers of samples from Encore™ samplers to preservative solution are performed within 48 hours of collection.

2.8 Unknown concentration samples, targeted for low level analysis are screened to determine if analysis should be performed using low level or medium level techniques.

2.9 All samples are introduced to a purge and trap device equipped with an auto sampler capable of aliquoting and transferring samples without affecting their integrity during sample transfer.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

The reporting and method detection limits are determined by the analytical method.

4.0 DEFINITIONS

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank; Instrument Blank, Storage Blank, and Sulfur Blank.

ENCORE™ SAMPLER - An inert polymeric container designed to hold approximately 5 or 10 grams of samples that can be sealed to prevent loss of volatile organic constituents.

MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOW, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method

blank is used to define the level of laboratory, background and reagent contamination.

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105°C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

PRESERVATIVE SOLUTION - Purge and trap grade methanol, Dl water or aqueous sodium bisulfate solution. Sediment samples from Encore™ samplers are placed into these solutions to prevent the loss of volatile organic constituents.

REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest.

SURROGATE SPIKE COMPOUND - A compound with chemical and physical characteristics similar to target analytes but not likely to be present in field samples that is spiked into all samples.
Surrogate accuracy is used to assess method performance on an individual field samples basis.

INSTRUMENT BLANK - a system evaluation sample containing lab reagent grade water with internal standards and surrogate standards added. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the SGS-Dayton Chemical Hygiene Plan which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 The following analytes covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, and vinyl chloride. Primary standards of these toxic compounds must be prepared in a hood. A NIOSH/Mass approved toxic gas respirator must be worn when the analyst handles high concentrations of these toxic compounds.

6.0 INTERFERENCES

6.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

6.2 Samples may become contaminated through contact with improperly cleaned field sampling equipment, contaminated preservative solution, and contaminated sample containers. Each of these items must be checked prior to use to assure that they are contaminant free of the compounds of interest.

6.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from reagent water or methanol carried through the sampling and handling protocol can serve as a check on such contamination.

6.4 Cross contamination can occur whenever high level and low-level samples are within the same proximity during transportation or storage. Contamination of this type cannot be affectivity controlled beyond the precautions normally employed for transportation and storage. However, the potential for cross contamination must be monitored through the analysis of field, trip, and storage blanks. Corrective action is taken if these blanks indicate the presence of contamination.

7.0 APPARATUS AND MATERIALS

7.1 Syringe

7.1.1 10cc graduated nylon syringe (Becton & Dickenson or equivalent).

7.1.1.1 Modify syringe by removing the tip at the zero (0) cc mark. The modified device is an open cylinder with plunger.

7.1.1.2 1 ml calibrated glass syringe (Hamilton or equivalent)

7.2 Sampling Vials

7.2.1 Container - 40 ml glass screw-cap VOA vial with Teflon-faced silicone septum, commercially purchased, pre-cleaned to USEPA specifications, documented to be analyte free.

7.2.2 Container - 2 ounce glass with phenolic resin cap and Teflon lid liner, commercially purchased, pre-cleaned to USEPA specifications, documented to be analyte free.

7.3 BALANCES

7.3.1 Analytical balance capable of weighing 0.0001 gram.

7.3.2 Top-loading balance capable of weighing 0.1 g.

7.4 Spatula

7.4.1 Disposable wood spatula sticks.

7.4.2 Stainless steel spatula.

7.5 Magnetic Stir Bars

7.5.1 12x4 mm (disposable)

7.6 Encore™ SAMPLERS

7.6.1 Encore™ Sampler - En Novative Technologies, Green Bay, Wisconsin

7.6.2 Encore™ Sample Extruder - En Novative Technologies, Green Bay, Wisconsin

7.6.3 Encore™ Coring Sampler - En Novative Technologies, Green Bay, Wisconsin

7.7 PLIERS

7.7.1 Hardware grade for removal of stubborn Encore caps.

7.8 BEAKERS - 250 ML

7.9 50 ml CLASS A GRADUATED CYLINDER

7.10 50 ml Class A Volumetric Flasks

8.0 REAGENTS AND STANDARDS

8.1 Preservative Solutions

8.1.1 Methanol: Pesticide quality or equivalent. Store apart from other solvents.

8.1.1.1 Methanol vials are prepared for sampling. 10mls of methanol is placed into 40ml VO vial and stored in a designated refrigerator. The vial is labeled with the preparation date, lot number of the methanol, a vial number and initials of the technician.

8.1.1.2 The vials are stored in a designated refrigerator in the lab and may be kept in-house up to 28 days after the preparation date. After 28 days they must be discarded.

8.1.1.3 Methanol vials are also purchased commercially. The manufacturers expiration date is used for those vials.

8.1.2 Sodium Bisulfate Solution:

8.1.2.1 Sodium bisulfate, NaHSO4 - ACS reagent grade or equivalent, add one (1) gram to 5ml reagent water.

8.2 Reagent water

8.2.1 Reagent water is defined as water in which an interferant is not observed at the method detection limit of the parameters of interest.

8.2.2 Reagent water is generated by either passing tap water through a bed of approximately one pound of activated carbon or by using the water purification system at the lab that is a series of deionizers and carbon cartridges.

9.0 SAMPLE COLLECTION, HANDLING, PRESERVATION, AND HOLDING TIME

9.1 SAMPLE COLLECTION

9.1.1 Sample Collection In Encore™ samplers.

9.1.1.1 Open the individual Encore™ sampler container package

9.1.1.2 Push down on the piston in the Encore™ unit until it is flush with the sampler opening.

9.1.1.3 Inset the sampler into the sample collection core device.

9.1.1.4 Insert the core device and sampler into the collected sample, taping gently. The piston will begin to move as sample fills the coring device.

9.1.1.5 The sampler is filled once the O-ring on the piston is visible in through the siting hole of the coring device.

9.1.1.6 Remove the sampler from the coring device, placing the cap firmly on the open sampler.

9.1.1.7 Place the unit in the Zip-Lock bag (provided), label, ice, and transport to the laboratory.

9.1.2 Low-level sample collection in DI water.

9.1.2.1 Initial bottle preparation for low level sampling is conducted in the laboratory as follows:

9.1.2.1.1 Add a pre-cleaned disposable magnetic stirring bar to a 40 ml purgeable vial.

9.1.2.1.2 (Optional) Add 1 gram of sodium bisulfate to the vial (0.2 g for each I gram of sample). Sodium Bisulfate preservation is only used for certain clients.

9.1.2.1.3 Add 5 ml of deionized water to the vial and seal using the screw cap and septum seal.

9.1.2.1.4 Add an adhesive label to the vial

9.1.2.1.5 Weigh the vial to the nearest 0.1 grams and record the tare weight on the bottle label. The bottle is now field ready.

9.1.2.2 Using the modified syringe, collect approximately 5 grams of sample (approx 4cc).

9.1.2.3 Remove the cap and place the tared vial on a calibrated toploading balance. Tare the balance to zero grams.

9.1.2.4 Carefully transfer approximately 5 grams of soil to the vial. Wipe any residual soil sample from the threads of the 40 ml vial. Immediately cap the vial, label, ice, and transport to the laboratory.

9.1.3 Medium level sample collection in methanol solution.

9.1.3.1 Initial bottle preparation for medium level sampling is conducted in the laboratory as follows:

9.1.3.1.1 Add 5mlml of methanol to a 40 ml purgeable vial and seal using the screw cap and septum seal.

9.1.3.1.2 Add an adhesive label to the vial as described in Section 8.1 .

9.1.3.1.3 Weigh the vial to the nearest 0.1 gram and record the tare weight on the bottle label. The bottle is now field ready.

9.1.3.2 Using the modified syringe, collect approximately 5g of sample (approx 4cc).

9.1.3.3 Remove the cap and place the tared vial on a calibrated toploading balance. Tare the balance to zero grams.

9.1.3.4 Carefully transfer approximately 5 grams of soil to the vial. Wipe any residual soil sample from the threads of the 40 ml vial. Immediately cap the vial, label, ice, and transport to the laboratory.

9.1.4 Sample Container Array. Sample containers must be assembled using the following per sample array:

9.1.4.1 Low Level or Unknown (<200ug/kg)

9.1.4.1.1 Field Preservation containers required if samples are targeted for low level analysis but may require medium level analysis because of high concentrations. Additional Encore™ units provide backup if sample re-analysis is required

9.1.4.1.2 2 x 40 ml vials (optionally containing NaHSO4) 1 x 40 ml vial containing CH3OH, 1 x 40 ml vial containing sample only for moisture determination, 1 x Modified Syringe

9.1.4.1.3 Quality Control samples only required if sample is low level: 1/Sample Group, up to 20 Samples, 2 x 40 ml vials containing Dl water.

9.1.4.1.4 Lab Preservation. Encore™ units are opened in the laboratory and preserved using the same procedures used for field preservation.

9.1.4.1.5 3 x Encore™, 1 x 40 ml vial containing sample only for moisture determination.

9.1.4.1.6 Quality Control: 1/Sample Group, up to 20 Samples, 2 x 40 ml vials (optionally containing $NaHSO_4$)

9.1.4.2 Medium Level (>200ug/kg)

9.1.4.2.1 Field Preservation

9.1.4.2.2 1 x 40 ml vial containing CH3OH, 1 x 40 ml vial containing sample only for moisture determination, 1 x Modified Syringe

9.1.4.2.3 Lab Preservation. Encore™ units are opened in the laboratory and preserved using the same procedures used for field preservation.

9.1.4.2.4 1 x Encore™, 1 x 40 ml vial containing sample only for moisture determination.

9.2 Sample Preservation & Transfer.

9.2.1 Field Preservation.

9.2.2 Samples collected in methanol must be transported and stored at < 6°C until analysis.

9.2.2.1 Samples for low level analysis are preserved in 5 ml of DI water (optionally 5 ml of sodium bisulfate).

9.2.2.2 Samples for medium level analysis are preserved in 5ml of methanol.

9.2.2.3 Vials that are purchased commercially contain 10mls of methanol.

9.2.3 All samples collected in Encore™ samplers must be transferred to a vial containing preservative solution within 48 hours of collection.

9.2.3.1 Upon receipt of samples, the chain of custody is reviewed for sampling date and time information to determine the 48-hour Encore holding period.

9.2.3.2 The samples are processed through login, assigned a sample number and a refrigerator location code.

9.2.3.3 A two-part label is generated with the smaller part of the label being placed on the Encore sampling bag and the larger part of the label tucked into the encore-sampling bag. The larger part of the label is applied to the volatile vial containing preservative solution after sample transfer.

9.2.3.4 Copies of the chain of custody are distributed to the Manager of GC/MS, the GC/MS scheduler, and a mail bin marked Encore samples.

9.2.4 Samples collected in methanol or sodium bisulfate solution must be stored at ^6°C and protected from exposure to light until analyzed. Samples collected in DI water are frozen at - 10°C and protected from light until analyzed. Certain regulatory agencies such as Ohio VAP do not allow freezing, therefore sodium bisulfate preservation must be used.

9.2.4.1 Samples received in methanol, DI water, or sodium bisulfate solution are logged in using standard login procedures.

9.2.4.2 The sample vial is weighed before adding the sample label. The weight is recorded in a logbook on the same entry line as the tare weight of the sample vial prior to sampling. Copies of the login information and the weights are distributed to GC/MS and to the computer log-in area.

9.2.4.3 The samples are then placed in the volatile soil refrigerator in storage at <6°C or in a freezer at -10°C awaiting the laboratory preparation procedure or analyses.

9.2.5 Samples collected in Encore™ units must be transported and stored at < 6°C until transferred to a preservative solution.

9.2.6 Intact Soil Samples

9.2.6.1 New York State allows the analysis of intact volatile soil samples without further preservation, unless client specific criteria state otherwise.

9.2.6.2 SGS-Dayton has determined that all other intact soil samples must be preserved within 48 hours of collection, as follows:

9.2.6.2.1 Upon receipt of the samples, the chain of custody is reviewed, the samples are processed through login, assigned a sample number, refrigerator location and the “SLL” code (soil-low level) is documented on the COC. The samples are then stored in the assigned refrigerator.

9.2.6.2.2 A copy of the COC and the necessary labels are delivered to a mail bin marked Encore samples.

9.2.6.2.3 Two low level samples will be prepared in DI water and frozen until analysis. One medium level sample will be prepared in methanol and stored at < 6°C until analysis.

9.3 Holding Time

9.3.1 Samples collected in Encore™ units are transferred to preservative solution within 48 hours of collection. The freezing process must be started within 47.5 hours, but will not be flagged as out of regulatory acceptance unless the freezing starts after the 48 hour time window from collection.

9.3.2 All samples are analyzed within 14 days of sampling unless otherwise specified by the contract.

9.4 STORAGE

9.4.1 Samples collected in Encore™ units must be stored at < 6°C until transferred to a preservative solution.

9.4.2 Samples preserved in methanol or sodium bisulfate solution must be stored at ^ 6°C and protected from exposure to light until analyzed. Samples preserved in Dl water must be frozen at -10°C and protected from exposure to light until analyzed.

9.4.2.1

10.0 PROCEDURE

10.1 Encore Handling Procedure

10.1.1 Copies of the chain of custody are distributed to the Manager of GC/MS, the GC/MS scheduler, and a mail bin marked Encore samples.

10.1.1.1 The Encore technician checks the mail bin regularly for the arrival of samples collected in Encore devices.

10.1.1.2 All samples collected in Encore™ samplers must be transferred to a vial containing preservative solution within 48 hours of collection.

10.1.1.3 If Encore units are awaiting transfer to preservative solution, the technician verifies that the units are within the 48-hour holding period.

10.1.1.4 The technician retrieves the samples listed on the chain of custody from the holding refrigerator and reconciles them against the chain of custody.

10.1.2 Samples are placed in preservative solution as follows:

10.1.2.1 Three(3) Encores received = two (2) low level, one (1) medium level.

10.1.2.2 Two(2) Encores received = one (1) low level, one (1) medium level.

10.1.2.3 One (1) Encore received = one (1) medium Level.

10.1.3 Low Level Laboratory Preservation.

10.1.3.1 Add a pre-cleaned disposable magnetic stirbar to a 40 ml purgeable vial.

10.1.3.2 Add 5ml of Dl water (optionally sodium bisulfate preservative solution) to the vial.

10.1.3.3 Add an adhesive label to the vial

10.1.3.4 Weigh the vial to the nearest 0.1 grams and record the tare weight on the bottle label.

10.1.3.5 Place the Encore™ sampler into the extruder tool.

10.1.3.6 Remove the cap from the Encore™ sampler.

10.1.3.7 Carefully transfer the entire contents of the Encore™ sampler to the vial containing the preservative solution. Wipe any residual soil sample from the

threads of the 40 ml vial. Read and record the weight of sample added to the vial. Immediately cap the vial, label, and freeze at -10°C or store at ^ 6°C (if sodium bisulfate is used) until analysis.

10.1.3.8 If the sample effervesces upon transfer to the sodium bisulfate preservative, discard the mixture and transfer a fresh Encore™ into a pre-weighed 40 ml vial containing 5ml of deionized water and a pre-cleaned disposable magnetic stirring bar. Follow steps 10.1.3.3-10.1.3.6. Store the vial in a freezer at -10°C until analysis. Preservation by freezing is not allowed by some regulatory agencies, such as Ohio VAP.

10.1.4 Medium Level Laboratory Preservation (transfer of sample from Encore™ samplers to preservative solution).

10.1.4.1 Add 5 ml of methanol to a 40 ml purgeable vial and seal using the screw cap and septum seal.

10.1.4.2 Add an adhesive label to the vial.

10.1.4.3 Weigh the vial to the nearest 0.1 grams and record the tare weight on the bottle label.

10.1.4.4 Place the Encore™ sampler into the extruder tool.

10.1.4.5 Remove the cap from the Encore™ sampler.

10.1.4.6 Carefully transfer the entire contents of the Encore™ sampler to the vial containing the preservative solution. Wipe any residual soil sample from the threads of the 40 ml vial. Read and record the weight of sample added to the vial. Immediately cap the vial, label, and store at < 6°C until analysis.

10.1.5 Intact Soil Sample Preservation

10.1.5.1 Methanol preservation, 1 vial.

10.1.5.2 Add 5ml of Methanol to a 40ml vial and seal using the screw cap and septum seal.

10.1.5.3 Add an adhesive label to the vial.

10.1.5.4 Weigh the vial to the nearest 0.1 gram and record the tare weight on the bottle label.

10.1.5.5 Remove the cap from the original sample container and transfer a portion (about 5 grams) of the sample into the vial. Wipe any residual soil sample from the treads of the 40ml vial. Read and record the weight of sample added to the vial. Immediately cap the vial, label and store at0- 6°C until analysis.

10.1.6 Dl water preservation, 2 vials.

10.1.6.1 Add a pre-cleaned disposable magnetic stirring bar to a 40 ml purgeable vial.

10.1.6.2 Add 5ml of DI water to the vial.

10.1.6.3 Add an adhesive label to the vial

10.1.6.4 Weigh the vial to the nearest 0.1 grams and record the tare weight on the bottle label.

10.1.6.5 Remove the cap from the Original sample container and carefully transfer a portion of the sample(about 5 grams) into the vial containing the DI water. Wipe any residual soil sample from the threads of the 40 ml vial. Read and record the weight of sample added to the vial. Immediately cap the vial, label, and freeze at - 10°C until analysis.

10.2 Autosampler Loading Procedure

10.2.1 All aqueous samples are screened prior to analysis. Prior to loading, the screen data for each sample is evaluated to determine if dilution is required prior to analysis.

10.2.1.1 Screening procedures are described in SOP GC3810.

10.2.1.2 The concentration of analytes detected during screening are evaluated to determine if the highest concentration compounds are within the calibration range of the instrument.

10.2.1.3 If the screen data indicates that samples exceed the calibration range, the highest concentration peaks are divided by the mid point calibration standard concentration to determine the dilution factor. This value is rounded to the next logical value to facilitate sample dilution as in the following example:

10.2.1.4 Screen Concentration (ug/l)/Mid Point Calibration Std (ug/l) = D.F.

10.2.1.4.1 4700 ug/l / 50 ug/l = 94 or 1:94, Use dilution of 1:100

10.2.1.5 Transfer the sample to be diluted to a 50 ml Class A volumetric flask with a calibrated syringe. Fill the volumetric flask to the volume mark with deionized water.

10.2.1.5.1 As in the example in 10.2.1.4, Transfer 0.5ml sample to the 50ml volumetric and fill to the volume mark with D.I. water to affect a 1:100 dilution.

10.2.1.6 Transfer the diluted sample to a 40 ml sampling vial for analysis.

10.2.2 Diluted and undiluted samples in 40ml sampling vials are sequenced by order of analysis and loaded onto the carousel of the auto sampler.

10.2.2.1 All specifications for sample analysis using purge and trap techniques are detailed in the individual SOPs for methods SW846 8015D (06/2003), SW846 8021 (12/96) and SW846 8260D (06/18).

11.0 QUALITY CONTROL

11.1 Field Blanks. Prepare aqueous field blanks for each field-sampling episode. Field blanks are analyzed in the laboratory to monitor for cross contamination during sampling.

11.2 Trip Blanks. Trip blanks are prepared to accompany every sample cooler. These blanks are used to determine if cross contamination occurred during sample transportation.

11.2.1 Aqueous trip blanks will be prepared for all low-level samples and all Encore™ samplers.

11.2.2 Methanol trip blanks will be prepared for all medium level samples collected using methanol field preservation techniques.

11.3 Method Blanks. Method blanks are prepared daily by the volatile prep lab to demonstrate that the soil preservative procedure, the instrument and reagents are free from interfering contaminants at the reporting limit of the analysis. Method blanks are prepared in the same manner as field soil samples with the exception of the addition of sample itself.

11.3.1 The following method blank configurations are prepared daily prior to the preparation of any field sample preservation or kit preparation,

11.3.1.1 Low Level Soil —

11.3.1.1.1 Prepare a low level method blank by pumping 5 ml of deionzied water from the dispenser used for field sample preservation.

11.3.1.1.2 Label the vial with the matrix type (Dl H20), prep date and time, and dispenser ID.

11.3.1.1.3 Deliver the prepared blank to the instrument designated by section supervisor.

11.3.1.1.4 Additional blanks may be prepared if needed.

11.3.1.2 Medium Level Soil - 5ml and 10ml dispensers

11.3.1.2.1 Prepare one medium level method blank from the selected dispenser.
11.3.1.2.2 Label each vial with matrix type (MEOH), prep date and time, dispenser ID, and lot #.
11.3.1.2.3 Deliver the prepared blank to the instrument designated by section supervisor.

11.3.2 The results of the method blanks should be less that the detection limit for all target compounds.

11.3.2.1 If any target compounds are detected a new blank is prepared and analyzed.

11.3.2.2 If the new method blank prepared from the replacement vials has no target compounds detected, then the soil prep procedure is assumed to be in control and the blank is evaluated as an instrument procedure blank.

11.3.2.3 If target compounds are detected in the replacement prep blank the section supervisor must be notified immediately to suspend use of the dispensing device in question. The dispensing device is cleaned and the solvents replaced until a satisfactory method blank is attained. Any samples prepared from a contaminated dispenser are re-prepared or footnoted accordingly as per the supervisors' discretion.

11.4 Matrix Spike Duplicates. A matrix spike duplicate pair (MS/MSD) is required to be analyzed for every twenty samples analyzed. The spike is prepared at the midpoint of the calibration range of the determinative method and is detailed in the SOP for that method.

11.4.1 MS/MSD for low concentration analysis. MS/MSDs for samples preserved in the field must be collected in duplicate. MS/MSDs for samples in Encore samplers must be collected in duplicate.

11.4.1.1 Select the Encore duplicate pair collected for quality control purposes and disperse the sample into a pre-weighed 40 ml purge vial containing either DI water or sodium bisulfate solution depending upon which is used for the sample.

11.4.1.2 Re-weigh the sample vial and record the weight of the sample.

11.4.1.3 Store either at -10°C or < 6°C (if sodium bisulfate is used) and protected from exposure to light until analyzed.

11.4.1.4 The prepared aliquot is spiked with the appropriate concentration of the matrix spike solution.

11.4.2 MS/MSD for MediumConcentration Analysis. Use methanol aliquots from the preserved sample to prepare the MS/MSD. Spiking of the methanolic extract is conducted at the instrument.

11.4.2.1 Dilute 1 ml of methanol solution to 50 ml of DI water in a 50 ml volumetric flask. Spike the flask with the matrix spiking solution. Prepare duplicate aliquots of this solution. Program the autosampler to spike the sample with surrogates and internal standards according to the requirements of the determinative method.

11.5 Spike Blank: A Spike Blank will be prepared and analyzed every batch of 20 samples. The Spike Blank recovery must meet the criteria specified in the individual analytical methods.

12.0 CALCULATIONS

12.1 Dilution Factor (DF). Dilution factors must be calculated for all medium level samples preserved with methanol. Calculate as follows:

$$DF = \frac{V_e \times V_a \times g}{G_s}$$

Where:
DF = Dilution Factor
V_e = Volume of Methanol Extract
V_a = Volume of Aqueous Extract
G_s = Grams Sample Extracted
g = units

12.2 Percent Solids

$$\%S = \frac{W_D}{W_W} \times 100$$

Where:
%S = Percent Solids
W_D = Dry Weight
W_w = Wet Weight

13.0 POLLUTION PREVENTION & WASTE MANAGEMENT

13.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 12.2.

13.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

13.3 Non-hazardous aqueous wastes.

13.4 Hazardous aqueous wastes

13.5 Chlorinated organic solvents

13.6 Non-chlorinated organic solvents

13.7 Hazardous solid wastes

13.8 Non-hazardous solid wastes

14.0 DOCUMENTATION

14.1 A preservation logbook of samples collected in Encore samplers must be maintained as a record of the date that samples are transferred to preservative solutions.

Current Version Revision Information

Changes / Edits made (this should include added or deleted information within a sentence or paragraph only):

Section / Subsection	Detailed description of what was revised
All	Accutest changed to “SGS-Dayton” or “the lab”
1.3, 10.2.2.1	Updated method revisions for 8015 and 8260
9.1.3.1.1	5 ml of methanol is added, not 10.
9.2.2.2	Medium level are also preserved with5 ml of methanol.
11.4.1.4	More detail added

Sections or Subsections deleted:

Section / Subsection	Reason section or subsection was removed

Sections or Subsections added:

Section / Subsection	Reason section or subsection was added
8.1.1.3	Methanol Vilas are also purchased commercially.
9.2.2.3	Purchased vials used for sampling contain 10ml.

History of Revisions

Version #	Date of Revision	Revised By
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END OF DOCUMENT

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Appendix D

Request To Import/Reuse Fill Material Form

NEW YORK STATE
DEPARTMENT OF ENVIRONMENTAL CONSERVATION

Request to Import/Reuse Fill or Soil

This form is based on the information required by DER-10, Section 5.4(e) and 6NYCRR Part 360.13. Use of this form is not a substitute for reading the applicable regulations and Technical Guidance document.

SECTION 1 – SITE BACKGROUND

The allowable site use is: Choose an item

Have Ecological Resources been identified? Choose an item

Is this soil originating from the site? Choose an item

How many cubic yards of soil will be imported/reused? Choose an item

If greater than 1000 cubic yards will be imported, enter volume to be imported:

SECTION 2 – MATERIAL OTHER THAN SOIL

Is the material to be imported gravel, rock or stone? Choose an item

Does it contain less than 10%, by weight, material that passes a size 100 sieve? Choose an item

Is this virgin material from a permitted mine or quarry? Choose an item

Is this material recycled concrete or brick from a DEC registered processing facility? Choose an item

SECTION 3 - SAMPLING

Provide a brief description of the number and type of samples collected in the space below:

Example Text: 5 discrete samples were collected and analyzed for VOCs. 2 composite samples were collected and analyzed for SVOCs, Inorganics & PCBs/Pesticides.

If the material meets requirements of DER-10 section 5.4(e)5 (other material), no chemical testing needed.

SECTION 3 CONT'D - SAMPLING

Provide a brief written summary of the sampling results or attach evaluation tables (compare to DER-10, Appendix 5):

Example Text: Arsenic was detected up to 17 ppm in 1 (of 5) samples; the allowable level is 16 ppm.

If Ecological Resources have been identified use the "If Ecological Resources are Present" column in Appendix 5.

SECTION 4 – SOURCE OF FILL

Name of person providing fill and relationship to the source:

Location where fill was obtained:

Identification of any state or local approvals as a fill source:

If no approvals are available, provide a brief history of the use of the property that is the fill source:

Provide a list of supporting documentation included with this request:

The information provided on this form is accurate and complete.

_____ _____

Signature Date

_____

Print Name

_____

Firm

Revised April 2023

Appendix E

Best Management Practice Plan (BMPP)

Town of Islip

Best Management Practices Plan

Long Island MacArthur Airport (ISP)
Ronkonkoma, NY 11779

January 2023

Best Management Practices Plan

Long Island MacArthur Airport (ISP)
Ronkonkoma, NY 11779

January 2023

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Our Ref:
30142977.BMPP.2023



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Version Control (optional)

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By

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Acronyms

AD	Aircraft Deicing/Anti-icing
AF	Aircraft Fueling
AM	Aircraft Maintenance
ANG	Air National Guard
AS	Aircraft Sanitary Service
AST	Aboveground Storage Tanks
BFF	Bulk Fuel Farm
BGM	Building and Grounds Maintenance
BMP	Best Management Practices
BMPP	Best Management Practices Plan
CBS	Chemical Bulk Storage
CS	Chemical Storage
CWA	Clean Water Act
DMR	Discharge Monitoring Report
EF	Equipment Fueling

ELG	effluent limitation guidelines
EM	Equipment Maintenance
FAA	Federal Aviation Administration
FBO	fixed base operators
FFF	firefighting foam
FH	Food Handling and Operations
FS	Fuel Storage
FT	Firefighting Foam Testing
gpm	gallons per minute
ISP	Long Island MacArthur Airport
MSDS	Material Safety Data Sheets
NPDES	National Pollutant Discharge Elimination System
NSPS	new source performance standards
NYSDEC	New York State Department of Environmental Conservation
PBS	Petroleum Bulk Storage
PH	petroleum hydrocarbons
POTW	Publicly Owned Treatment Works
R-TD	Runway and Taxiway Deicing/Anti-icing
RCP	Reinforced Concrete Pipe
SCDHS	Suffolk County Department of Health Services
SIC	standard industrial classification
SPCC	Spill Prevention, Control, and Countermeasure
SPDES	State Pollutant Discharge Elimination System
SRP	Spill Prevention Response
TOI	Town of Islip
USEPA	United States Environmental Protection Agency
UST	Underground Storage Tank
VF	Vehicles Fueling
VM	Vehicle Maintenance

1 Introduction

This guidance document represents the operational Best Management Practices Plan (BMPP) for the Long Island MacArthur Airport (ISP). It has been compiled to facilitate ISP compliance with the requirements of the New York State Department of Environmental Conservation (NYSDEC) State Pollutant Discharge Elimination System (SPDES) program. This BMPP is intended for use by Town of Islip (TOI) Department of Transportation and Aviation and ISP tenants to provide consistent and effective management of stormwater runoff quality. The BMPP presents a description of the facility and a discussion of potential pollutant sources resulting from practices and activities at the airport. This BMPP also identifies existing stormwater management controls and Best Management Practices (BMPs) at the airport and identifies BMPs that reduce or eliminate pollutants entering the stormwater drainage system.

Tenants shall employ all practicable BMPs and always comply with applicable local, state, and federal regulations and conform to the ISP Rules and Regulations as well as the requirements of all applicable agreements pertaining to contractor, occupant, and tenant activities.

ISP currently holds an individual industrial SPDES Permit (Permit No. NY 0173703). The BMPP was prepared as a guidance document for airport tenants and ISP employees to adhere to the ISP's individual SPDES Permit. A copy of the individual SPDES Permit is included in **Appendix A**.

1.1 Regulatory Background

This section presents an overview of the regulatory history of the stormwater pollution control program at the federal and state levels.

1.1.1 Federal Requirement

The Federal Aviation Administration (FAA) imposed more stringent requirements on deicing activities to increase the margin of safety for aircraft operating during snow and ice conditions. These new protocols are grounded in the "clean aircraft concept", which requires that aircraft surfaces must be free of snow and ice before departure and imposes time limits (holdover time) between the application of deicing fluids and aircraft departure. These revised FAA protocols (FAA, 1992) have increased the quantities of deicing fluids used by U.S. airlines and airports. This increase in deicing activity, along with increased air traffic, has resulted in greater quantities of deicing fluid being entrained in airport runoff. Consequently, each year large quantities of propylene glycol-based aircraft deicing fluids are used to de-ice aircraft.

In 1972, the Federal Water Pollution Control Act, also known as the Clean Water Act (CWA), was enacted to require that the discharge of pollutants to waters of the United States from any point source be covered by a National Pollutant Discharge Elimination System (NPDES) permit. In 1987, amendments to the CWA added Section 402(p), establishing a framework for regulating municipal and industrial discharges of stormwater under the NPDES program. The NPDES program is administered by the United States Environmental Protection Agency (USEPA).

The NPDES regulations require operators of specific types of industrial activities to discharge stormwater to obtain NPDES permits. Stormwater associated with industrial activities is defined as stormwater runoff that exits any conveyance that is used for collecting and conveying stormwater that is directly related to manufacturing,

processing, material storage, and waste material disposal areas and similar areas where stormwater can contact industrial pollutants related to the industrial activity at an industrial facility. The airport's standard industrial classification (SIC) code of 4581 was included as a regulated industry and therefore is required to obtain NPDES permits.

The USEPA under 40 CFR Part 449 proposed a new rule on August 28, 2009, titled "Effluent Limitation Guidelines and New Source Performance Standards for the Airport Deicing Category." The comment period closed on February 26, 2010. The USEPA promulgated the Airport Deicing Effluent Guidelines on May 16, 2012. The USEPA requires technology based effluent limitation guidelines (ELGs) and new source performance standards (NSPS) under the CWA for discharges from airport deicing operations. The requirements generally apply to wastewater associated with the deicing of aircraft and airfield pavement at primary commercial airports that have 1,000 or more non-propeller aircraft departures, and 10,000 or more total annual departures. Airports qualifying under this rule are required to collect at least 60 percent of spent aircraft deicing fluid and treat the wastewater. Airports must meet the numeric discharge requirements for Chemical Oxygen Demand (daily maximum of 271 mg/L and weekly average of 154 mg/L) and Ammonia as Nitrogen (daily maximum of 14.7 mg/L) at the location where the effluent leaves the onsite treatment system. They may either treat the wastewater on-site or send it to an off-site treatment contractor or Publicly Owned Treatment Works (POTW). The ELGs are incorporated into the NPDES permits issued by USEPA, states, or tribes.

1.1.2 State Requirement

The federal regulations also allow states to implement the NPDES program and issue NPDES permits to regulate stormwater discharges associated with industrial activities. The NYSDEC has been delegated authority by the USEPA to implement the NPDES program under the SPDES program.

1.2 SPDES Permit NY 0173703

NYSDEC issued ISP an Individual Stormwater Permit (SPDES No.: NY0173703)/ DEC Number 1-4728-00783/00003 with a Permit Effective Date on September 1, 2018, and Permit Expiration Date of August 31, 2028. The SPDES Permit authorizes discharge covers discharges from stormwater associated with ISP industrial activities to groundwater.

Outfall Monitoring

NYSDEC issued the SPDES Permit that authorizes stormwater discharge from the airport and requires the monitoring of Outfall Numbers 001 and 002. The monitoring (sampling/analysis) of these outfalls will be conducted by ISP. The outfalls will be monitored for parameters of interest based on the anticipated discharges from various activities at the airport.

Outfall 001 includes stormwater from tank farm area and discharges to groundwater. Outfall 002 includes stormwater from ramp area and discharges to groundwater. Outfalls 001 and 002 require monitoring of pH, oil and grease, benzene, toluene, xylene, and ethylbenzene. Outfall 002 also requires monitoring of total nitrogen, total phosphorus, and propylene glycol for propylene glycols associated with the airlines' deicing activities. **See Figure 1 in Attachment B for the locations of Outfalls 001 and 002.**

TOI and Tenant Responsibility

The NYSDEC issued the SPDES Permit to the TOI as the owner and operator of the ISP. Compliance with the SPDES Permit requires monitoring of Outfalls 001 and 002 such that there are minimal impacts to stormwater from airport activities. Tenants and/or other entities that perform activities that can contribute to pollutant sources resulting from practices and activities at the airport and apply or otherwise use deicing and/or anti-icing materials at the airport shall be required to comply with the SPDES Permit.

BMPP Implementation

This BMPP, as required by the SPDES Permit will discuss the current activities performed at the airport and provide guidance BMPs developed for areas occupied by tenants of the airport to help minimize activity-based impacts to stormwater. Tenants and/or other entities that apply or otherwise use deicing and/or anti-icing materials at the airport shall participate/review/adhere in the development of the BMPP.

SPDES Compliance

In addition to stormwater discharge monitoring, the Individual SPDES Permit requires the ISP to track and report any anticipated (e.g., planned releases) or unanticipated (e.g., spills or leaks) non-compliance with the SPDES Permit. The Individual SPDES Permit requires that any non-compliance with permit conditions be reported to NYSDEC. Anticipated non-compliance is defined as any planned releases and unanticipated non-compliance events include unanticipated bypass, any upset which violates any effluent limitation in the permit, violation of maximum daily discharge limitation, and spills or leaks. Reports for anticipated non-compliance should be filed with NYSDEC prior to the discharge.

1.3 SPDES Special Conditions - Best Management Practices

The activities conducted at ISP involve multiple tenants performing many varied tasks associated with routine airport operations. Examples of the activities performed at the airport with the potential to cause pollution of stormwater discharges include but are not limited to aircraft, vehicle and equipment fueling and maintenance, building and grounds maintenance, chemical and fuel storage, and deicing/anti-icing of aircraft, taxiways, and runways, vehicle/equipment washing, and lavatory service operations.

ISP has developed a comprehensive approach to managing stormwater discharges associated with activities at the airport, including identifying potential pollutants through conducting of routine facility inspections, distribution of compliance performance forms, and development of plans and specific best management practices to minimize and/or reduce potential pollutant discharges to groundwater.

Permit Conditions and Special Permit Conditions from Permit Effective Date on September 1, 2008, Permit Effective Date on September 1, 2013, and Permit Effective Date on September 1, 2018, are included and reference to BMP and BMPP section are noted below to ensure compliance during the terms of the SPDES Permit. Most recent Special Conditions are noted and where appropriate Special Conditions from previous permit is noted herein.

1.3.1 Special Condition – Number 1 General

Special Condition – BMPP, Page 4 of 9 of the SPDES Permit requires ISP to develop, maintain, and implement a BMPP to prevent releases of significant amounts of pollutants, including deicing/anti-icing chemicals, to the waters of the State through plant site runoff; spillage and leaks; sludge or waste disposal; and stormwater discharges including, but not limited to, drainage from raw material storage. The BMPP shall be documented in narrative form and shall include the 13 minimum BMPs and any necessary plot plans, drawings, or maps. Other documents already prepared for the facility such as a Safety Manual or a Spill Prevention, Control and Countermeasure (SPCC) plan or Spill Prevention and Response (SPR) plan may be used as part of the plan and may be incorporated by reference. A copy of the current BMPP shall be submitted to the NYSDEC as required in Special Condition 2 and a copy must be maintained at the facility and shall be available to authorized NYSDEC representatives upon request. The BMPs applicable to each area of the facility occupied by a particular tenant shall be consistent with an overall BMPP for the entire airport. The permittee shall maintain, update, and assure the proper implementation of the overall BMPP.

In accordance with Special Condition– Best Management Practices Number 1, TOI compiled this BMPP for ISP. The BMPP is intended for use by ISP and tenants to provide consistent and effective management of storm water runoff quality. The BMPP presents a description of the various facilities at ISP and a discussion of potential pollutant sources resulting from practices and activities at ISP. The BMPP also identifies existing storm water management controls and BMPs at the various facilities at ISP and identifies BMPs that reduce or eliminate pollutants entering the stormwater drainage system. The ISP BMPP identifies 15 BMPs to eliminate or reduce pollutants in storm water runoff:

BMP 1:	Aircraft Deicing
BMP 2:	Aircraft, Vehicle, and Equipment Fueling
BMP 3:	Aircraft, Vehicle, and Equipment Maintenance
BMP 4:	Aircraft, Vehicle and Equipment Washing, Steam Cleaning and Degreasing
BMP 5:	Building Cleaning and Maintenance
BMP 6:	Chemical and Petroleum Storage and Handling
BMP 7:	Elimination of Non-Storm water Discharges to Storm Drains
BMP 8:	Spills Management
BMP 9:	Oil/Water Separators
BMP 10:	Outdoor Handling of Material
BMP 11:	Outdoor Material and Equipment Storage
BMP 12:	Waste Management
BMP 13:	Stormwater Pollution Prevention Education
BMP 14:	Pavement Sweeping & Stormwater Facility Maintenance
BMP 15:	Security

The ISP BMPP has been provided to the tenants for review and implementation. TOI has reviewed facility components and systems to confirm industrial activities conducted at each of the tenant facilities. In addition, the TOI has conducted site visits and inspections to clarify facility components and systems, confirm the routine operations of some of the tenants, and confirm ongoing operations and the current use of BMPs by some of the tenants.

1.3.2 Special Condition – Number 2 Compliance Deadlines

Special Condition – BMPP, Page 4 of 9 of the SPDES requires ISP to implement and maintain a BMPP. The BMPP shall be reviewed annually and shall be modified whenever (a) changes at the facility materially increase the potential for release of pollutants' (b) actual release indicate the plan is inadequate; or (c) a letter from the Department identifies inadequacies in the plan. The permittee shall certify in writing, as an attachment to the Discharge Monitoring Report (DMR), that the annual review has been completed.

In accordance with Special Condition – Number 2, ISP prepared an Annual Site Compliance Evaluation in Fall 2022 and an updated BMPP in Winter 2023. A copy of the current BMPP was submitted to the NYSDEC as required in Special Condition 2 and a copy will be maintained at the facility and shall be available to authorized NYSDEC representatives upon request.

1.3.3 Special Condition – Number 3 Facility Review

Special Condition – Best Management Practices Number 3 on Page 4 of 9 of the SPDES Permit requires "the permittee to review and inspect all facility components or systems (including but not limited to material storage areas; in-plant transfer, process, and material handling areas; loading and unloading operations; storm water, erosion, and sediment control measures; process emergency control systems; and sludge and waste disposal areas) where material or pollutants are used, manufactured, stored or handled to evaluate the potential for the release of pollutants to the waters of the State. Tenants and/or other entities who apply or otherwise use deicing and/or anti-icing materials at the facility shall participate in this facility review and inspection. Tenants and/or other entities who apply or otherwise use deicing and/or anti-icing materials shall also provide all necessary information to the permittee for the permittee to complete its evaluation. In performing such an evaluation, the permittee shall consider such factors as the probability of equipment failure or improper operation, cross-contamination of storm water by process materials, settlement of facility air emissions, the effects of natural phenomena such as freezing temperatures and precipitation, fires, and the facility's history of spills and leaks. The relative toxicity of the pollutant shall be considered in determining the significance of potential releases. The review and inspection shall evaluate whether measures to reduce pollutant loadings identified in the BMPP are adequate and properly implemented in accordance with the terms of this permit or whether additional control measures are needed."

In accordance with Special Condition – Number 3, ISP prepared an Annual Site Compliance Evaluation in Fall 2022 for all ISP entities and tenants at ISP. The Annual Site Compliance Evaluation consisted of inspections and review of all ISP and tenant facility components and systems including; fueling stations, waste storage areas, above ground and underground storage tanks (e.g. petroleum, glycol and chemical storage), spills management and oil/water separators, loading areas, material handling area, deicing/anti-icing; which have the potential to discharge to one or more of the ISP outfalls listed in the SPDES Permit, covering six (6) drainage areas and evaluation of overall effectiveness of the BMPP.

The Annual Site Compliance Evaluation provides a basis for evaluating the overall effectiveness of the BMPP and measuring the BMPP performance. The Annual Site Compliance Evaluation also documents the date, time, inspection methodology, inspectors, BMP conformance evaluation results, and the overall effectiveness of the BMPP. Any proposed modifications to the inspection procedures and approach for BMP implementation will also be included with this report.

1.3.4 Special Condition – Number 4 13 Minimum BMPs

Special Condition – Best Special Condition – Best Management Practices, Number 4 on Page 4 of 9 of the SPDES Permit requires development of 13 minimum BMPs to prevent or minimize the potential release of pollutants to the waters of the State. **Section 4** of the ISP BMPP discusses the 13 minimum BMPs, including two (2) additional BMPs, that were developed using the September 1992 manual for *Storm Water Management for Industrial Activities*, EPA 832-R-92-006 as reference. Activity specific BMPs that are applicable to ISP activities are provided in **Appendix D**.

1.3.5 Special Condition – Number 5 Facilities with Petroleum and/or Chemical Bulk Storage (PBS and CBS) Areas

Special Condition – Best Management Practices, Number 5 on Page 4 of 13 of the SPDES Permit (Permit Effective Date 2013) requires that facilities with petroleum and/or chemical bulk storage areas must comply with all applicable regulations including those involving releases, registration, handling, and storage of petroleum and/or chemicals; stormwater discharges from handling and storage areas should be eliminated where practical. Tenants that store petroleum and hazardous chemicals must comply with all applicable regulations and prepare and maintain a Spill Prevention Control and Counter measure (SPCC) Plan and Spill Prevention and Response (SPR) Plan. ISP obtained tenant SPCCs and SPRs and incorporated information contained in those plans during the development of the BMPP

In accordance with Special Condition Number 5, Compliance must be maintained with all applicable regulations including those involving releases, registration, handling, and storage (6NYCRR 595-599 and 612-614). Stormwater discharges from handling and storage areas should be eliminated where practical.

1.3.5.1 Spill Cleanup

All spilled or leaked substances must be removed from secondary containment systems as soon as practical and for CBS storage areas within 24 hours, unless written authorization is received from the Department. The containment system must be thoroughly cleaned to remove any residual contamination which could cause contamination of stormwater and the resulting discharge of pollutants to waters of the State. Following spill cleanup, the affected area must be completely flushed with clean water three times and the water removed after each flushing for proper disposal in an on-site or off-site wastewater treatment plant designed to treat such water and permitted to discharge such wastewater. Alternately, the permittee may test the first batch of stormwater following the spill cleanup to determine discharge acceptability. If the water contains no pollutants, it may be discharged. Otherwise, it must be disposed of as noted above. See Discharge Monitoring below for the list of parameters to be sampled for.

1.3.5.2 Discharge Operation

Stormwater must be removed before it compromises the required containment system capacity. Each discharge may only proceed with the prior approval of the permittee staff person responsible for ensuring SPDES permit compliance. Bulk storage secondary containment drainage systems must be locked in a closed position except when the operator is in the process of draining accumulated stormwater. Transfer area secondary containment drainage systems must be locked in a closed position during all transfers and must not be reopened unless the transfer area is clean of contaminants. Stormwater discharges from secondary containment systems should be

avoided during periods of precipitation. A logbook shall be maintained on site noting the date, time and personnel supervising each discharge.

1.3.5.3 Discharge Screening

Prior to each discharge from a secondary containment system the stormwater must be screened for contamination*. All stormwaters must be inspected for visible evidence of contamination. Additional screening methods shall be developed by the permittee as part of the overall BMP Plan, e.g., the use of volatile gas meters to detect the presence of gross levels of gasoline or volatile organic compounds. If the screening indicates contamination, the permittee must collect and analyze a representative samples' of the stormwater. If the water contains no pollutants, it may be discharged. Otherwise, it must either be disposed of in an on-site or off-site wastewater treatment plant designed to treat and permitted to discharge such wastewater or the Regional Water Engineer can be contacted to determine if it may be discharged without treatment.

1.3.5.4 Discharge Monitoring

Unless the discharge from any bulk storage containment system outlet is identified in the SPDES permit as an outfall with explicit effluent and monitoring requirements, the permittee shall monitor the outlet as follows:

1.3.5.4.1 Bulk Storage Secondary Containment Systems:

(a) The volume of each discharge from each outlet must be monitored. Discharge volume may be calculated by measuring the depth of water within the containment area times the wetted area converted to gallons or by other suitable methods. A representative sample shall be collected of the first discharge' following any cleaned-up spill or leak. The sample must be analyzed for pH, the substance(s) stored within the containment area and any other pollutants the permittee knows or has reason to believe are present.

(b) Every fourth discharge* from each outlet must be sampled for pH, the substance(s) stored within the containment area and any other pollutants the permittee knows or has reason to believe are present**.

1.3.5.4.2 Transfer Area Secondary Containment Systems:

The first discharge' following any spill or leak must be sampled for flow, pH, the substance(s) transferred in that area and any other pollutants the permittee knows or has reason to believe are present**.

1.3.6 Special Condition – Number 5 SWPPP for Construction Activity

Special Condition – Best Management Practices, Number 4 on Page 5 of 9 of the SPDES Permit requires development of a Stormwater Pollution Prevention Plan (SWPPP) prior to the initiation of any site disturbance of one (1) acre or more of uncontaminated areas. The NYSDEC has prepared a new SPDES General Permit (GP-0-20-001) for Stormwater Discharges from Construction Activity. The GP-0-20-001 for Stormwater Discharges from Construction Activity became effective on January 29, 2020 and will expire on January 28, 2025.

Stormwater at MacArthur Airport is conveyed to catch basins that discharge to groundwater. According to SPDES General Permit (GP-0-20-001) a SWPPP is not required for discharges of stormwater from construction activity to groundwaters.

1.3.7 Special Condition – Number 6 Airports Additional Requirements for BMPP

1.3.7.1 Special Condition – Number 6.A.i Site Map

Special Condition – Best Management Practices, Number 6.A.i on Page 5 of 9 of the SPDES Permit requires a site map that will identify where any of the following activities may be exposed to precipitation/surface runoff: aircraft and runway deicing/anti-icing operations; fueling stations; aircraft, ground vehicle, and equipment maintenance/cleaning areas; and storage areas for aircraft, ground vehicles and equipment awaiting maintenance.

In accordance with Special Condition – Number 6.A.i., ISP prepared a site map identifying these activities which is provided in Appendix B. Section 3 identifies and describes all activities at ISP.

1.3.7.2 Special Condition – Number 6.A.ii Summary of Potential Pollutant Sources

Special Condition – Best Management Practices, Number 6.A.ii on Page 5 of 9 of the SPDES Permit requires a narrative description of the potential pollutants sources from the following activities: aircraft, runway, ground vehicle and equipment maintenance and cleaning; aircraft and runway deicing/anti-icing operations (including apron and centralized aircraft deicing/anti-icing stations, runways, taxiways, and ramps). Facilities which conduct deicing/anti-icing operations shall maintain a record of the types (including the Material Safety Data Sheets (MSDS)) and monthly quantities of deicing/anti-icing chemicals used, either as measures amounts, or in the absence of metering, as estimated amounts. This includes all deicing/anti-icing chemicals. Tenants and fixed-base operators who conduct deicing/anti-icing operations provide the above information to ISP which is included in the ISP BMPP.

In accordance with Special Condition Number 6.A.ii, Tenants provide ISP with monthly quantities of deicing/anti-icing chemicals used, the number of aircraft deiced during the event, and the date of deicing and/or anti-icing, in addition, tenants maintain MSDS sheets. These forms also gather information about the use of BMPs by tenants during the application of deicing/anti-icing materials.

Section 3 identifies and describes all activities and potential pollution sources of stormwater pollution at ISP.

1.3.7.3 Special Condition – Number 6.B.i Good Housekeeping

Special Condition – Best Management Practices, Number 6.B.i on Page 6 of 9 of the SPDES Permit requires a description and practices measures that prevent or minimize the contamination of stormwater runoff from various airport activities.

Section 4 discusses recommended BMPs that are applicable to ISP activities.

1.3.7.4 Special Condition – Number 6.B.ii Source Reduction

Special Condition – Best Management Practices, Number 6.B.ii on Page 6 of 9 of the SPDES Permit suggests alternatives to the use of glycol-based deicing/anti-icing chemical.

Section 4 discusses some alternatives to the use of glycol-based deicing/anti-icing chemical that are practiced that can reduce the aggregate amount of deicing/anti-icing chemicals used and/or lessen the environmental impact.

1.3.7.5 Special Condition – Number 6.B.iii Management of Runoff

Special Condition – Best Management Practices, Number 6.B.iii on Page 7 of 9 of the SPDES Permit indicates that ISP shall describe and implement a program to control to manage contaminated runoff to reduce the amount of pollutants being discharges from ISP. The stormwater management program shall describe the primary issues that are currently driving decisions regarding the types of deicing runoff for collection systems, recovery systems, and conveyance systems.

Section 4 discusses stormwater management to control and manage contaminated runoff to reduce the amount of pollutants being discharged from ISP.

1.3.7.6 Special Condition – Number 6.B.iv Routine Facility Inspections

Special Condition – Best Management Practices, Number 6.B.iv on Page 7 of 9 of the SPDES Permit indicates that routine facility inspections shall be specified in the ISP BMPP.

In accordance with Special Condition Number 6.B.iv, ISP prepared an Annual Site Compliance Evaluation in Fall 2022 to track and monitor BMPP effectiveness.

Sections 4 and 5 discuss the routine inspections conducted at the facility, inspection forms, a recommended schedule, a list of the pollution prevention team personnel, employee training requirements, facility inspection protocol, monitoring requirements, recordkeeping, and reporting procedures, BMPP updates, and comprehensive site compliance evaluation.

1.3.7.7 Special Condition – Number 6.B.v Comprehensive Site Compliance Evaluation

Special Condition – Best Management Practices, Number 6.B.v on Page 7 of 9 of the SPDES Permit indicates that an annual site compliance evaluation shall be conducted by qualified facility personnel during periods of actual deicing operations, if possible.

In accordance with Special Condition Number 6.B.v, ISP completed the Annual Site Compliance Evaluation in Fall 2022.

Section 5 discusses the facility inspection protocol and comprehensive site compliance evaluation that is conducted on an annual basis.

2 Airport Description

This section describes the location of ISP, current airport land uses, and the airport stormwater drainage system. In addition, a comprehensive list of the facilities and tenant activities has been included.

2.1 General

Long Island MacArthur Airport is at 100 Arrival Avenue Ronkonkoma, Suffolk County, NY 11779. The Town of Islip Department of Transportation and Aviation owns and operates the ISP. The airport is bound on the south by Route 454, Veteran's Highway, on the west by Smithtown Avenue, on the north by Railroad Avenue and on the east side by Lincoln Avenue as shown on **Figure 1 in Appendix B.**

The airport covers approximately 1,311 acres with a total of 65 facilities and 31 tenants. The airport incorporates three (3) runways, fourteen (14) taxiways, aircraft ramps, a main terminal building, parking lots, bulk fuel facilities and numerous individual buildings and aircraft hangars. The airport is serviced by several major air carriers and is also an active general aviation and corporate aviation facility serving the Long Island area. Typically, the airport operates 24 hours per day, seven (7) days per week and has a staff of 85 TOI employees.

The airport Standard Industrial Code (SIC) is 4582, "Air Transportation".

The TOI owns the following buildings at ISP:

1. Main Terminal and Baggage Area (Building #1)
2. Two vehicle maintenance facilities (Buildings #11 & #12).
3. Airport Fire and Rescue (Building # 5)
4. Bulk Fuel Farm (Building #21)
5. Whitney Hangar (Building # 20)
6. Aero Trades Hangar (Building # 53)
7. Security Outpost (Building # 66)
8. Town Hazmat Office (Building # 15)
9. Compost facility (Buildings #57, #58 & #59, which are excluded from this BMPP)

Major airport tenants currently include the FAA, Southwest Airlines, and Frontier Airlines, Breeze Airways plus fixed base operators (FBOs) who under license to ISP provide hangar, maintenance and fueling service to general aviation and corporate aircraft. In addition, there are several corporate hangars and miscellaneous aircraft support/maintenance service facilities. The Clarion Hotel is also on the airport property. An airport layout plan, including building numbers, is provided in **Figure 2 in Appendix B**.

2.2 Land Use

The airport can be characterized as commercial, surrounded by commercial properties to the north, south, and west, with residential areas to the east of the airport property. The airport occupies 1,311 acres and includes approximately 165 acres of impervious surfaces, or 14% of the total area. The nearest surface water is Lake Ronkonkoma which is approximately one (1) mile north northwest of the northwest corner of the airport.

2.3 Drainage System

Figure 3 in Appendix B is the current Airport Location Plan (ALP), indicating the major structures plus impervious and pervious areas of the airport. The airport occupies 1,311 acres and includes approximately 165 acres of impervious surfaces, or 14% of the total area.

The stormwater drainage network at ISP consists of six (6) drainage areas. Storm water from impervious surfaces drains either as surface sheet flow, or in buried storm drain lines, typically to either natural grassy swale areas or manmade recharge basins which vary from 20 to 50 feet deep. Since groundwater is 50 feet below grade, the deeper recharge basins have groundwater exposed.

Since none of the stormwater discharged at ISP enters surface water of the US, the discharge points will be defined as those points where point discharge of stormwater into the ground (and thus to the groundwater) occurs. Typically, this is via pervious grassy swale areas, or recharge basins serving large impervious area watersheds such as aircraft runways or ramps. Individual or multiple drywells are typically used to drain vehicle parking areas. The surface topography is relatively flat, ranging in elevation from 77 feet to 98 feet above sea level. The soil is typically sand or gravel, with scrub pine vegetation and the depth to groundwater is approximately 50 feet.

Figure 3 in Appendix B provides the drainage areas at ISP and the points of discharge (to groundwater) for each of these drainage areas and the apparent storm water drainage patterns. A description of the airport storm drainage system is provided below. **Table 1** presents the description of the characteristics of each drainage area.

Table 1. Stormwater Drainage Areas

Drainage Area	Description	Discharges to
A	Terminal Area & Southeast Side of Airport	Recharge Basin 1
B	General Aviation at West Side of Airport	Recharge Basin 2
C	Central, East Side Runways and Taxiways	Swale Area 3
D	Central, North Side Runways and Taxiways	Swale Area 4
E	General Aviation and South-Central Side of Airport	Recharge Basin 5
F	Southwest Corner, General Aviation, Taxiway & Runways	Swale Area 6

2.3.1 Drainage Area A

Drainage Area A is located on the southeast side of the airport and includes the terminal building, short term parking and the fueling and deicing ramp at the terminal. Paved aircraft and vehicle parking areas are affected by industrial activities and are therefore included in this BMPP. This watershed consists largely of impervious concrete or asphalt pavement, most of which involves industrial activity, and it drains via a network of buried storm drains to Recharge Basin 1, which is approximately fifty feet deep with groundwater constantly exposed. Some of the vehicle parking lots discharge stormwater directly via drywells. The pervious areas drain directly to groundwater as the soil is a sand gravel mix and stormwater does not remain on the surface after a storm event.

2.3.2 Drainage Area B

Drainage Area B is located on the west side of the airport and includes all the west side aircraft hangars and adjacent taxiways and runways. Paved aircraft and vehicle parking areas are affected by industrial activities and are therefore included in this BMPP. This watershed consists largely of impervious concrete or asphalt pavement, most of which involves industrial activity such as impervious hangar ramps, taxiways, and runways, which drain via a network of buried storm drains and open swales into a grassy recharge basin bounded by Runway 15R, Runway 6 and Taxiway W. Some of the vehicle parking lots discharge stormwater directly via drywells. The pervious areas drain directly to groundwater as the soil is a sand gravel mix and stormwater does not remain on the surface after a storm event. Because of the high permeability of the soils in the pervious areas, and the absence of site activities in such pervious areas, drainage from pervious areas will not be addressed further in this BMPP, unless they act as recharge basins.

2.3.3 Drainage Area C

Drainage Area C is located on the east side of the airport and is bounded by Taxiway E, Runway 33R, Runway 24, and the eastern property line of the airport. It includes these impervious areas plus large areas of grass and native trees. Buried culverts under taxiways and runways are used to direct stormwater into natural grassy swale areas. Because of the high permeability of the soils in the pervious area, and the relative absence of industrial activities, except for private hangars adjacent to the Civil Air Patrol building, drainage from this pervious area will not be addressed further in this BMPP.

2.3.4 Drainage Area D

Drainage Area D is located on the north side of the Airport and is bounded by Runway 15R, Runway 24, and Railroad Avenue on the northern property line of the Airport. It includes these impervious areas plus large areas of grass and native trees, and the Town of Islip Compost Facility plus the Long Island Railroad Ronkonkoma rail yard. Buried culverts under taxiways and runways are used to direct stormwater into natural grassy swale areas. Because of the high permeability of the soils in the pervious area and the relative absence of industrial activities (the Compost Facility and rail yard are not included in the Airport), drainage from this pervious area will not be addressed further in this BMPP.

2.3.5 Drainage Area E

Drainage Area E is located on the south-central side of the airport (between Taxiway S and the Terminal) and it includes all the south side aircraft hangars, ramps, the bulk fuel facility and one FBO, (Modern Aviation), the Air National Guard (ANG) helicopter facility plus rental car facilities and the Airport maintenance facility. This watershed consists largely of impervious concrete or asphalt pavement, most of which involves industrial activity such as hangar ramps where fueling and deicing takes place, and rental car fueling and maintenance. Some of the vehicle parking lots discharge stormwater directly to groundwater via drywells. The pervious areas drain directly to groundwater as the soil is a sand gravel mix and stormwater does not remain on the surface after a storm event. Because of the high permeability of the soils in the pervious areas, and the absence of site activities in such pervious areas, drainage from pervious areas will not be addressed further in this BMPP, unless they act as recharge basins.

2.3.6 Drainage Area F

Drainage Area F is located on the southeast corner of the airport (between Taxiway S, Runway 6 and Veterans Memorial Highway) and it includes one FBO (Mid Island Air Service) the airport bulk fuel facility the south side aircraft hangars, ramps, the bulk fuel facility, and the Holiday Inn hotel. This watershed consists largely of impervious concrete or asphalt pavement, most of which involves industrial activity such as the bulk fuel facility, hangar ramps where fueling and deicing takes place. Some of the vehicle parking lots discharge stormwater directly to groundwater via drywells. The pervious areas drain directly to groundwater as the soil is a sand gravel mix and stormwater does not remain on the surface after a storm event. Because of the high permeability of the soils in the pervious areas, and the absence of site activities in such pervious areas, drainage from pervious areas will not be addressed further in this BMPP, unless they act as recharge basins.

Average annual rainfall at ISP is 47 inches and a 25-year one hour rainstorm is 2.5" and a 25-year, 24-hour storm is 5.8".

2.4 Historical Stormwater Monitoring

ISP performs monthly monitoring of Outfalls 001 and 002.

2.5 Tenants and Target Industrial Activities

ISP's tenants conduct a variety of industrial activities supporting the airport's operations with the potential to impact the quality of stormwater runoff at the airport.

Site inspections, interviews, and past environmental plans were reviewed to determine potential stormwater runoff impacts at the tenant facilities. Based on this information, the following activities that occur at the airport pose a potential to impact stormwater quality at ISP:

- Aircraft Deicing/Anti-Icing (AD)
- Aircraft Fueling (AF)
- Aircraft Maintenance (AM)
- Aircraft Rental/Sales (AR)
- Outdoor Storage Areas (OS)
- Chemical Storage (CS)
- Equipment Degreasing (ED)
- Vehicle Fueling (VF)
- Equipment Storage (ES)
- Fuel Storage (FS)
- Floor Washdown (FW)
- Aircraft Painting (AP)
- Aircraft Washing (AW)
- Grounds Maintenance (GM)
- Runway Maintenance (RM)
- Equipment Fueling (EF)
- Vehicle Washing (VW)
- Equipment Maintenance (EM)

Appendix D provides recommended BMPs for each of these Target Industrial Activities. For purposes of this BMPP, the term "vehicles" includes mobile equipment powered by petroleum-based fuels (e.g., trucks, luggage tugs, etc.) while "equipment" is used to describe other non-mobile equipment (e.g., generators). A more specific discussion of tenant activities and the potential pollution sources related to the activities is presented in Section 3.

3 Identification of Potential Pollution Sources

This section identifies and describes all activities and potential sources of stormwater pollution at ISP. Specifically, potential stormwater pollutants, areas of potential pollutant contact with stormwater, activity-based non-stormwater discharges, potential hard-piped non-stormwater discharges, and historic spills and leaks were explored through the BMPP questionnaire, onsite interviews, and a thorough visual inspection.

Historical data and environmental audits were used to facilitate interviews with tenants. The tenants were asked to provide the following information:

- General description of operations conducted at each of the tenant's locations;
- Facility stormwater drainage patterns;
- Specific target activities conducted at the facility, and whether they are performed indoors or outdoors;
- Inventory of chemicals at each location and chemical storage practices;
- Identification of potential pollution sources;
- Identification of any existing best management practices;
- Identification of non-stormwater discharges and possible illicit connections to the stormwater drainage system;
- Description of deicing operations; and
- Identification of historic leaks and spills.

3.1 Non-Stormwater Discharges

Several sources of non-stormwater discharges are possible at ISP. Non stormwater discharges include small spills from aircraft, equipment, vehicle fueling and washing, and firefighting foam (FFF). Aircraft deicing during dry weather can also lead to non-stormwater discharges. Frost can occur on airplane wings during dry weather when surrounding air temperatures reach the "frost point" where water vapor in the air sublimates directly to ice and coats the wing. Deicing does not occur at ISP airport during non-deicing months and therefore is not a source of stormwater discharge during that period. However, if deicing were required or if deicing were to occur during the non-deicing months, the deicing entity would be responsible for preventing discharges to the stormwater systems.

Firefighting foam testing is not performed at ISP. Firefighting foam can be a potential pollutant during emergency situations where uncontrolled discharge could migrate to the stormwater drainage system. If firefighting foam is discharged during an incident, it will be treated as a spill and cleaned up to the maximum practicable extent to minimize the potential for pollutant migration into the stormwater drainage.

3.2 Activity Specific Potential Pollution Sources

A brief description of the activities with the greatest potential to be discharged into the stormwater drainage system and the areas in which they are performed is provided below.

3.2.1 Aircraft, Vehicle, and Equipment Maintenance

Chemicals such as lubricating oils, hydraulic oils, fuels, degreasers, and other cleaning products are routinely used in airport maintenance activities. Small leaks and spills are not uncommon during maintenance activities; therefore, the potential for pollutant contact with stormwater is greatly increased when these activities are performed outdoors. This potential is further increased if these outdoor activities are performed near stormwater drains. Generally, indoor areas only present a potential for pollutant contact with stormwater if floor drains discharge to the stormwater drainage system.

Major aircraft maintenance such as engine overhauls and repair are not performed at ISP. The air carriers perform some light aircraft maintenance at ramps including adding oil and changing oil, tires, and lights. Tenants are required to use drip pans when performing maintenance such as adding oil or changing oil at the ramps. Tenants are educated on the potential impacts and risks of pollutants entering the drainage system because of maintenance activities. The small spills of chemicals and petroleum hydrocarbons that occur during these limited aircraft maintenance operations are typically cleaned up using granular absorbent materials. There is a potential that residuals from small spills in these areas can become entrained in the overland flow of stormwater runoff and thus drained to catch basins through a Reinforced Concrete Pipe (RCP) lateral varying in size from 6-inch to 24-inch. These laterals connect to the main RCP which then increases in size up to 42-inch. Once the main RCP leaves the terminal area the RCP increases in size up to 72 inch as it enters Recharge Basin #1.

Major vehicle and equipment maintenance is not performed at ISP, however some minor maintenance activity to vehicles and equipment does occur at several tenant locations. Based on the extent to which these activities are performed, they present a low potential for pollutant contact with stormwater.

3.2.2 Aircraft, Vehicle, and Equipment Fueling

Aircraft and vehicle fueling is performed at various locations throughout the airport property. Aircraft fueling is performed outdoors at the ramp locations at the passenger terminal, the FBOs and corporate hangars. The greatest concern with aircraft and vehicle fueling is the potential for minor spills, which usually originate from topping-off or overfilling of aircraft and vehicles. The major constituents of aircraft and vehicle fuels are petroleum and hydrocarbons. These minor spills can be entrained in the overland flow of stormwater runoff and transported into the stormwater drainage system.

ISP tenants routinely perform aircraft fueling at ramps and no aircraft fueling occurs indoors. Vehicle fueling occurs outdoors at the locations indicated above. Considering the large volume of aircraft and vehicle fueling performed outdoors at ISP, these activities present significant potential for pollutant contact with stormwater.

3.2.3 Aircraft, Vehicle, and Equipment Washing

Aircraft washing in outdoor areas is not allowed at ISP due to Suffolk County Department of Health Services (SCDHS) Article 7 which imposes restrictions in groundwater aquifer areas. Washing operations must ensure that all wastewater is captured and taken off site for disposal. No vehicle wash water may enter storm drains or pervious areas. None of the tenants at ISP perform outdoor aircraft washing.

No vehicle wash water discharges into the storm drains. Vehicle washing that is typically associated with the car rental areas occurs within a designated area on the leasehold and is part of a 100% recycle, zero discharge system.

Considering that these operations are either performed indoors or that all wash water is collected for disposal via off site when performed in outdoor areas, these activities present a low potential for pollutant contact to stormwater.

3.2.4 Aircraft Sanitary/Lavatory Service

Aircraft sanitary service operations involve connecting a hose from the tank on a lavatory truck to the aircraft's lavatory facilities and emptying the contents into the truck. The contents are disposed of offsite. Minor spills may occur during the connection and disconnection of the hose onto and off the aircraft. Personnel are instructed to position a "Waste Catch Bucket" under the lavatory service panel. There is a potential that biocides, bacteria, and other pollutants from these minor spills can become entrained in the overland flow of stormwater runoff and be transported to the stormwater drainage system.

Due to the low volume of aircraft sanitary services performed on ISP airport property and the observation of routine minor spills, this activity presents a moderate potential for pollutant contact with stormwater.

3.2.5 Aircraft Deicing/Anti-Icing

Deicing/anti-icing operations protect runways, taxiways, and aircraft from accidents, which can result from ice and snow build-up on runways, taxiways, and aircraft during inclement weather. Deicing is performed as necessary at ISP during the winter season, (November through March). The deicing/anti-icing season may fluctuate based on local weather conditions and other weather conditions around the country. Aircraft, runways, taxiways, aprons are deiced or anti-iced at the airport.

Aircraft deicing removes snow, ice, or frost from the aircraft via the use of propylene glycol or a blend of propylene glycol with water. Aircraft deicing/anti-icing activities are performed by tenants. Deicing agent is sprayed onto the aircraft from a tank truck. The ratio of glycol to water in Type I deicing fluid varies from tenant to tenant. Type IV anti-icing fluid is usually applied without dilution. Application ratios vary depending on use and weather conditions.

Ice and snow are typically removed from aircraft using a heated mixture of Type I deicing fluid and hot water applied under pressure. Undiluted Type I fluids must contain a minimum of 80 percent propylene glycol by weight, with the balance composed of water, buffers, wetting agents, and oxidation inhibitors. Deicing chemicals used in aircraft deicing must also be non-corrosive to prevent damage to aircraft aluminum and sensitive electronic systems. Type I deicing fluid is applied at gate areas at the passenger terminal.

Aircraft anti-icing may follow deicing to prevent the further accumulation of snow or ice on the deiced surfaces either while aircraft are waiting for take-off during especially severe weather, or during overnight parking. Anti-icing is accomplished by applying Type IV anti-icing fluid to clean (i.e., ice free) aircraft surfaces. Type IV anti-icing fluids are also composed of propylene glycol, along with thickeners that allow the fluid to cling to the aircraft and provide prolonged protection and longer holdover times. This increased viscosity is lost when Type IV anti-icing fluid undergoes shear stresses, such as those experienced during takeoff. The unique characteristics of Type IV anti-icing fluid require that special low-shear applicator nozzles be used. In addition, because Type IV fluid adheres to the aircraft until an air speed of approximately 85 knots is reached, it is widely dispersed through sloughing during taxiing and takeoff. Also because of this property, Type IV fluid is only used on larger aircraft that reach a rotational speed of more than 85 knots during takeoff. Anti-icing is conducted at the same locations as deicing.

Propylene glycol (Type I or Type IV) is stored in aboveground storage tanks (ASTs) adjacent to the passenger terminal. The aircraft deicing vehicles fill up at this storage area and then the aircraft deicing normally occurs at the gates at the passenger terminal. Primary deicing occurs within one hour of takeoff.

At ISP, air carrier deicing takes place on the aircraft terminal apron. The deicing and anti-icing fluid (currently propylene glycol diluted with water).is stored in AST on the air side of the Terminal Building, and deicer trucks are used to transport, heat, and apply the deicing fluid to aircraft. Stormwater runoff from this ramp is collected from a series of catch basins by an underground concrete pipe which discharges into Recharge Basin A, located northeast of the Terminal Buildings.

The discharge of aircraft deicing fluid / stormwater contaminated with propylene glycol deicing fluid to groundwater within a drinking water aquifer is a regulated discharge under the current SPDES permit, and groundwater monitoring wells down gradient of Retention Basin A are used to monitor the impact of deicing activities on the aquifer.

Overspray of deicing/anti-icing fluids and drip and shear of deicing/anti-icing fluids during takeoff and landings have the potential to impact stormwater runoff. Deicing material that falls off the aircraft during deicing is collected in catch basins and transported through the airport's storm drain system. Deicer that remains on the aircraft after application either drips off while taxiing to a runway or shears off the aircraft during takeoff. Deicer that shears off the aircraft either falls onto the runway or infield or is dispersed as small droplets into the air.

All the tenants at ISP now use propylene glycol and the use of ethylene glycol is strongly discouraged. Potential pollutants from deicing/antic-icing activities include propylene glycol. ISP airport is required to maintain a record of the types (including the material safety data sheets (MSDS)) and monthly quantities of deicing/anti-icing chemical used. Tenants who conduct deicing/anti-icing operations shall provide the above information to airport staff for inclusion in the ISP BMP for the entire facility. MSDSs for deicing/anti-icing chemicals used at ISP airport and the monthly usage of these chemicals shall be provided to the airport.

The use of deicing chemicals presents a high potential for contamination of stormwater during winter months. Figure 4 located in Appendix B indicates the location of deicing activities and treatment at ISP.

Considering the volume of aircraft and number of runways and taxiways at ISP and that the deicing activities are performed outdoors prior to or during precipitation events these activities present significant potential for contact with stormwater. The airport installed an aircraft deicing fluid pretreatment system to process all aircraft deicing fluid captured in the terminal ramp drainage system during an aircraft deicing event prior to discharge to groundwater.

3.2.6 Runway and Taxiway Deicing/Anti-Icing

Runway, taxiway, ramp, and apron deicing is the responsibility of the TOI. The purpose of pavement deicing/anti-icing is to break the bond holding ice and snow to the surfaces of runways and taxiways, facilitating mechanical ice and snow removal also to maintain adequate friction between aircraft tires and the runway. Residual deicing materials on the pavement provide anti-icing protection.

Runway, taxiway, and ramp deicing normally utilize sodium formate, and/or potassium acetate. Typically, airport runways are sprayed with deicing agents and anti-icing agents, in extreme icing conditions only, to prevent the buildup of ice and snow that would inhibit , takeoff, and landing. Sand is typically used to prevent slippage at the gate area.

In addition, sand/salt mixtures are used at ISP to improve traction at the terminal roadway system, pedestrian traffic areas, and parking areas when necessary. The sand applied in the terminal area is collected by sweepers once the pavement has dried. Sand applied to the terminal roadway system generally is washed to the edge of the pavement, is trapped by the soils and vegetation of the infield areas, or travels to the storm drain system. ISP uses visual observation to determine the frequency and severity of grit or sand/salt application applied to maintain traction, adjusting as necessary, consistent with flight and road traffic safety.

Spent deicing fluid has the potential to contaminate groundwater or surface water it flows off airport runways to storm drains or to waterways as sheet runoff.

3.2.7 Fuel Storage

Six (6) 50,000-gallon jet fuel ASTs, which contain aircraft Jet-A fuel, are located at the facility's Bulk Fuel Farm (BFF) which is owned by the airport. These six (6) tanks are used for commercial aircraft operations. In addition, the BFF has two (2) 30,000-gallon underground storage tanks (USTs) for Jet-A and one (1) 12,000-gallon UST for avgas, for use by FBOs. These tanks have been approved by the SCDHS, Office of Pollution Control. The responsibility for this function has been delegated to this agency by the NYSDEC under the Petroleum Bulk Storage (PBS) regulations.

Each tank is equipped with high level alarm, inventory monitoring and tank overfill systems. This high-level system activates alarms and shutdown procedures to prevent tank overfilling.

Concrete dikes surround the BFF ASTs, with 150 percent containment capacity. This 150 percent containment can hold the contents of a single largest tank plus sufficient freeboard to allow for precipitation. The dike at the BFF is equipped with drain valves. Stormwater collected in the diked area is drained into a pretreatment facility. This facility has an oil water separator to scavenge floating oil store it. The effluent from the oil water separator is pumped through a liquid phase carbon filter prior to discharge to one of two (2) 20,000-gallon stormwater USTs. The full UST is sampled and analyzed by a laboratory for compliance with SPDES requirements prior to discharge at outfall #001.

Any discharge of BFF containing stormwater must comply with Permit Limits on Page 3 of 9 of the SPDES Permit, a copy of which is provided in **Appendix A**.

The BFF is operated by Swissport who maintain an SPCC Plan which includes appropriate spill prevention and clean up measures.

In addition, the airport has petroleum tanks located at:

- Main Terminal and Baggage Area (Building #1)
  - 2,000-gallon AST, boiler back up fuel
- Vehicle maintenance facilities (Buildings #11 & #12)
  - 5,000-gallon UST for diesel fuel
  - 5,000-gallon UST for gasoline
  - 2,000-gallon UST for gasoline
  - 1,000-gallon UST for waste oil
  - Two (2) 275-gallon ASTs for engine oil and hydraulic fluid

3.2.8 Chemical Storage

ISP airport maintenance facilities typically store paints and thinners in stockrooms, parts cleaning fluids at its automotive garages, and anti-icing and deicing glycol at TOI and tenant areas. Chemicals stored outdoors at the ISP airport include propylene glycol; all other chemicals are located indoors. Chemicals are stored in double walled tanks located on concrete slabs with absorbent material available to facilitate cleanup or in secondary containment to capture any spills. Chemicals stored indoors present a low potential for pollutant contact with stormwater.

3.2.9 Building and Grounds Maintenance

Most of the building and ground maintenance at ISP is performed by the TOI. Several tenants subcontract building maintenance services to outside professionals. Pesticides and herbicides are used to maintain aprons, runways, and building green spaces. During rainfall events, accumulated residues from pesticide and herbicide usage can encounter stormwater and be transported into the stormwater drainage system.

ISP's licensed maintenance staff perform pesticide and herbicide applications to service landscaped areas. Due to the limited areas of non-indigenous plants at the airport property this activity presents a moderate potential for pollutant contact with stormwater.

3.2.10 Outdoor Storage Activities

Raw materials, by-products, leaking equipment and vehicles, and containers exposed to stormwater at outdoor storage areas can adversely impact stormwater runoff.

There are numerous vehicle and equipment storage areas throughout ISP airport. Several tenants store gasoline and/or diesel fuel on airport property. Most of this storage is located outdoors and is primarily comprised of ASTs, drums, and stationary tanker trucks. Materials stored outdoors at ISP airport include jet fuel, gasoline, diesel, waste oil, lubricating oil, propylene glycol, and aircraft and vehicle detergents. Ground support equipment and vehicles are also parked or stored outdoors at various locations throughout ISP airport. Fluids (e.g., fuel oil, propylene glycol, hydraulic fluid, chemical toilet water, and deicing fluids) leaking from vehicles can adversely impact stormwater runoff, Potential pollutants from outdoor storage activities include oil and grease, petroleum hydrocarbons, metals, volatile organic compounds, and propylene glycol.

Fifty-five (55) gallon storage drums are typically used at ISP airport to store virgin and waste material. Many of these drums are in interior maintenance areas and as such they are not being exposed to stormwater. Under SCDHS regulations, no floor drains are allowed in indoor storage areas as they would form potential release pathways.

Considering the aircraft and ground vehicle fueling and fuel truck staging areas and the frequency with which minor spills occur, this activity presents a significant potential for stormwater pollutant contact.

3.3 Summary of Pollutant Contact with Stormwater Potential

As part of the Annual Site Compliance Evaluation prepared for ISP to evaluate the effectiveness of BMP implementation and performance, the potential for pollutant contact from the industrial activities above was re-evaluated and is presented in Table 2 below. Table 2 serves to determine if additional BMPs are required or if BMP modifications are needed for any of the target industrial activities at ISP because of increased potentials for pollutants to encounter stormwater identified during the review of the Annual Site Compliance Evaluation.

Table 2: Pollutant Contact with Stormwater Potential

Activity Specific Potential Pollutant Sources	2016 BMPP	2022 Annual Site Compliance Evaluation	2023 BMPP
Aircraft, Vehicle, and Equipment Maintenance	Low	Based on Number of incidents	Low
Aircraft, Vehicle, and Equipment Fueling	High	Based on Number of Spills/Leaks	Moderate
Aircraft, Vehicle, and Equipment Washing	Low	Based on Number of incidents	Low
Aircraft Sanitary/Lavatory Service	Moderate	Based on Number of incidents	Moderate
Aircraft Deicing/Anti-Icing	High	Based on use	High
Runway and Deicing/Anti-Icing	Low	Based on use	Low
Fuel/Equipment Storage	Moderate	Based on Number of spills/leaks	Moderate
Chemical Storage	Low	Based on Number of incidents	Low
Building and Grounds Maintenance	Moderate	Based on Number of incidents	Moderate
Outdoor Storage Activities	Moderate	Based on Number of incidents	Moderate

Based on the evaluation of the Annual Site Compliance Evaluation to determine BMP effectiveness, much of the identified pollution contact with stormwater potential stayed constant. The results of this evaluation confirm that BMPs are effective in controlling the potential for pollutants from industrial activities to enter the stormwater, and that no additional BMPs are needed for these activities.

3.4 Potential Pollution Sources and Pollutants of Concern

Many of ISP's tenants perform activities in areas that present the potential for stormwater pollutants to be discharged into the stormwater system. The tenant activities with the greatest potential to contribute to stormwater pollution and some of the contaminants of concern for each activity are listed below:

Aircraft Deicing/Anti-icing (AD)

- Glycols
- Sodium acetate
- Potassium acetate

Aircraft, Vehicle, and Equipment Maintenance (AM, EM, VM)

- Oil and grease
- Petroleum hydrocarbons
- Propylene Glycol
- Halogenated Solvents
- Non-halogenated Solvents

Aircraft, Vehicle, and Equipment Fueling (AF, EF, VF)

- Petroleum hydrocarbons
- Fuel Vapors

Aircraft Sanitary Service (AS)

- Biocides
- Bacteria

Building and Grounds Maintenance (BGM, PH)

- Pesticides
- Herbicides
- Oils and grease
- Petroleum Hydrocarbons

Chemical Storage (CS) and Fuel Storage (FS)

- Petroleum hydrocarbons
- Lubricants
- Paints
- Battery Acid
- Solvents

Food Handling Operations (FH)

- Oil and grease
- Bacteria

Firefighting Foam Testing (FT)

- Volatile organic compounds, Per- and Polyfluoroalkyl Substances (PFAS)

Runway and Taxiway Deicing/Anti-icing (R-TD)

- Glycols
- Sodium acetate
- Potassium acetate

The potential pollutants that may be discharged into the stormwater drainage system from a tenant location are based on the activities conducted by the tenant. The pollutants consist primarily of petroleum products (such as fuels, oils, and greases), halogenated and non-halogenated solvents, and deicing fluids (propylene glycol), oils, greases, petroleum hydrocarbons, solvents, and glycols from aircraft, equipment, and vehicle fueling, maintenance, and washing activities are potential pollutants because the activities are generally performed outdoors in proximity to storm drains. Pollutants from these activities potentially can be transported to the stormwater drainage system either as direct spills (dry weather flow) or from rainfall runoff that mobilizes residual contaminants (wet weather flow).

3.5 Hard-Piped Non-Stormwater Discharge Identification

ISP staff is not currently aware of any existing hard-piped, non-stormwater discharges with potential to discharge into the existing stormwater drainage system. Further, in the event new or suspected hard-piped, non-stormwater discharges are discovered, they will be dye/smoke tested for confirmation and eliminated/sealed as needed. Hard-piped illicit connections are defined as equipment that discharges directly to the stormwater drainage system. These could include oil/water separators, interior floor and trench drains, utility sinks, and chiller and boiler overflow/blow down lines.

3.6 Runoff and Erosion Management

There is little potential for erosion problems at ISP since most of the airport is impervious. The stormwater drainage system is a closed RCP conduit that discharges directly into Recharge Basins. The principal potential source for erosion at ISP can occur during construction activities. Construction projects of any size should be reviewed before the project begins to determine if adequate soil and erosion control procedures will be implemented.

3.7 Spill Reporting and Leaks

A list of reportable quantity spills and leaks of toxic or hazardous pollutants that have occurred during the past three (3) years at areas that are exposed to precipitation or that otherwise drain to a stormwater conveyance at ISP airport are recorded. Each act of non-compliance shall include a short description of the non-compliance, a description of any actions and mitigation taken or proposed by the TOI or tenant entity to limit environmental impact associated with the non-compliance. The TOI or tenant shall provide an estimate of the date when the corrective or mitigation action will be completed.

3.8 Stormwater Monitoring and Sampling

Stormwater monitoring and sampling data provides information on the quality of stormwater runoff at ISP airport. The stormwater analytical data is used to identify the types and sources of pollutants and to provide a means for

evaluating the environmental risk for stormwater runoff, ISP is subject to various stormwater monitoring and sampling as required by Special Condition 1, Page 3 of 9 of the SPDES Permit.

4 Storm Water Management Controls

4.1 General

A stormwater BMP is defined as any program, technology, process, siting criteria, operating method, measure, or device that controls, removes, or reduces pollution. The permit requires the development and implementation of BMPs to address pollutants originating from industrial sources. Appropriate BMPs for industrial facilities were selected based on a site reconnaissance, tenant interviews, and information obtained from tenant questionnaires. Areas of actual or potential pollutant contact are evaluated and applicable BMPs are recommended or implemented to eliminate or minimize the potential for discharge of stormwater pollutants. BMPs can be classified into categories based on whether the intended stormwater control objective is quality control or quantity control.

Quality control BMPs are designed to limit the types and concentrations of pollutants found in stormwater runoff and are subdivided into source control BMPs and treatment control BMPs. Source control BMPs are operational practices intended to prevent pollutants from entering surface waters by altering performance of activities to eliminate or minimize pollution produced because of the activity. Source control BMPs generally involve eliminating a target activity's exposure to stormwater and typically include the following:

- Moving an outdoor operation indoors;
- Covering an outdoor activity or storage area with either a roof or a lean-to;
- Placing chemical or petroleum storage containers in a shed or under a lean-to;
- Storing hazardous materials/wastes in covered, contained areas.

A properly designed and implemented spill response program can also be an effective method for protecting stormwater quality. Spill response programs rely upon employee awareness and training to be effective.

Treatment control BMPs are a type of quality control BMP that treats stormwater to remove pollutants. Examples of treatment BMPs include:

- Oil/water separators;
- Grass swales;
- Retention basins;
- Infiltration; and
- Filtration.

Quantity control BMPs are intended to control the runoff volume or peak discharge rate. The use of stormwater detention basins is one example of a quantity control BMP. A properly designed and maintained detention basin can also decrease the amount of pollutants entering surface waters, thereby improving receiving water quality.

Section 2.5 described industrial activities typically performed by ISP tenants. A more detailed discussion of potential pollution sources is described in Section 3. Activities performed indoors have less potential to affect runoff water quality, although practices such as hosing down indoor floor space to outdoor areas after performing industrial activities contradict the potential water quality benefits of performing industrial activities under cover.

ISP tenants perform industrial activities directly related to aviation, such as aircraft operation, maintenance, and cargo handling, as well as general industrial activities such as vehicle maintenance, equipment storage, and facility maintenance. Many of the ISP tenants have already implemented a variety of acceptable BMPs to

minimize the effects of these activities on stormwater quality. The following sections supplement and enhance current BMP implementation and provide for consistent airport-wide application.

4.2 Good Housekeeping Practices

Good housekeeping practices that address housekeeping, operational, structural, and contingency considerations that can be taken to help achieve environmental compliance are utilized at ISP. The purpose and intent of good housekeeping is to minimize the exposure of pollutants to rainfall and runoff, The following practices are the basis of a good housekeeping program and are the minimum acceptable at ISP:

- Walkways, aisles, roadways, and exits are always to be kept clear.
- Small spills are to be cleaned up immediately and disposed of in an approved manner.
- Refuse is to be placed in an appropriate container.
- Material and products are stored in a neat and orderly fashion with particular attention not to block walkways or access routes.
- Chemical containers are to be stored in enclosed or covered areas whenever possible to minimize contact with stormwater.
- Chemical storage containers are to be properly labeled.
- Empty drums are to be placed only in their designated area.
- Chemical containers and/or drums are always to be kept closed when not in use.
- Inside floors are to be kept clear of debris and spills and are to be swept or mopped regularly.
- Tools and equipment are to be kept clean and neatly stored when not in use.

4.3 Minimum Best Management Practices

The maximum benefit of a stormwater pollution prevention program can be achieved only if tenants implement the BMPs that correspond to the target industrial activities performed at their facilities. Special Condition - Best Management Practices, Number 4 on Page 4 of 9 of the SPDES Permit requires development of 13 minimum BMPs to prevent or minimize the potential release of pollutants to the waters of the State.

4.3.1 Pollution Prevention Team

Individual tenants shall have designated personnel responsible for implementing the BMPs at corresponding tenant sites. Table 3 lists the members of the airport Pollution Prevention Team (PPT) for all facilities covered under the ISP BMPP. This list will be updated when a change to the personnel occurs. It is the responsibility of each facility to notify the ISP BMPP coordinator or their designated representative when there are personnel changes. Other PPT member responsibilities include ensuring implementation of appropriate BMPs, retaining a copy of the implemented BMPP onsite, and providing feedback to the ISP BMPP coordinator or a representative regarding BMPP compliance.

Table 1. Airport Pollution Prevention Team

Name	Title	BMPP Function
Robert Schneider	Deputy Commissioner	BMPP Executive
Al Cinotti	Town of Islip – Chief of Fire Rescue	BMPP Coordinator

The BMPP Coordinator for the facility is Al Cinotti (631-467-3279) and his duties include the following:

The BMPP Coordinator will implement this BMP for all TOI operated facilities and will inspect each tenant facility to ensure that they are following the SPDES permit requirements. Each tenant facility will be surveyed to confirm conformance with the requirements of the airport BMPP. In addition, the airport will conduct regular facility and stormwater drainage system inspections for both airport and tenant operated facilities to ensure that the required BMPs have been implemented and monitored by the tenants in accordance with the requirements of the BMPP.

4.3.2 Reporting of BMP Incidents

All spills that occur on airport property are to be reported to and logged by the BMPP Coordinator. These spills will be added to the spill history and kept with the BMPP. The BMPP coordinator will document the chemical spilled, location of spill, quantity spilled, date and time, corrective action taken and whether the spill resulted in a non-stormwater discharge. Tenants shall be individually responsible for reporting all spills to the appropriate regulatory agency.

Tenants are required to notify TOI and Fire Rescue with a spill of any size identifying the event date/time, location, type of spill, response, gallons spilled, cause/reason for spill or leak, and recommended remedial action.

The Fire Rescue team will respond to the incident to ensure the spill is being handled correctly. In the event the air carrier cannot handle the release TOI will mitigate the spill with the staff of ISP Fire Rescue and Maintenance.

If ISP Fire Rescue and Maintenance are unable to handle the spill, TOI contacts the TOI Haz-Mat Response Team and if further assistance is needed TOI will request assistance from the Suffolk County Strike Team.

4.3.3 Risk Identification & Assessment

Identify and determine the potential pollutant sources from each tenant and their leasehold. This will entail gathering information, at a minimum, with respect to the type of activities performed at their site, the type of materials stored which potentially can contribute pollution to stormwater runoff from ISP. Based on this information the most appropriate BMPs are selected to prevent or control pollutants from these areas.

4.3.4 Employee Training

The ISP BMPP Coordinator shall make available a copy of the BMPP and offer a BMPP implementation training for PPT members. These members in turn are to train their own staff. Training will cover items such as prohibited discharges, inspections, spill response, good housekeeping, implementation of BMPs, deicing activities and record keeping procedures.

4.3.5 Inspections and Records

As part of the Annual Site Compliance Evaluation, qualified facility personnel are to inspect designated equipment and areas of the airport on an annual basis to assess the effectiveness of BMP implementation and the overall BMPP. All records should be kept for at least three (3) years after the termination of the existing permit.

4.3.6 Security

Proper security measures are taken to prevent unauthorized access to secure areas at ISP. Heightened procedures have been implemented as an extra precaution. Criminal background checks, carry-on bags are searched more carefully, and passengers' names are cross-checked with lists of people the Federal government deems suspicious. Security systems are in place to prevent an accidental or intentional release of material to stormwater runoff, include training security personnel, lighting, and access control. ISP security measures require all current and prospective employees who have access to secure areas of airports to undergo fingerprinting and criminal history background checks.

4.3.7 Preventative Maintenance

A preventative maintenance program involves routine inspection and maintenance of structural BMPs (i.e., cleaning oil/water separators, catch basins) as well as inspecting and testing facility equipment and systems to identify and correct conditions that could cause breakdowns or failures, which could potentially result in the discharge of pollutants to the stormwater drainage system. This includes identifying equipment and areas to inspect and development of a schedule for routine inspections. The prompt repair or replacement of defective equipment found during inspections and testing should be performed and documented. The most appropriate BMPs are selected to prevent or control pollutants from these areas which potentially can contribute pollution to stormwater runoff from ISP.

4.3.8 Good Housekeeping

Good housekeeping requires routine maintenance of equipment and chemical storage areas in a clean and orderly manner to reduce the likelihood of contaminating stormwater runoff. Basic operation and maintenance BMPs incorporated into a Good housekeeping program include maintenance conducted on the terminal apron and in dedicated hangars. The following practices are part of the good housekeeping program:

- performing maintenance activities indoors;
- maintaining an organized inventory of material used in the maintenance areas;
- draining all parts of fluids prior to disposal;
- preventing the practice of hosing down the apron or hangar floor;
- using dry cleanup methods, and
- Collecting stormwater runoff from the maintenance area and providing treatment or recycling.

4.3.9 Materials/Waste Handling, Storage, and Compatibility

Employees should be trained in which materials are hazardous, where they are stored, how they are labeled, and in their proper use. A program has been established to identify material, promote waste reduction and recycling. Establish designated storage areas and segregate waste. Communicate recycling requirements and instructions.

4.3.10 Spill Prevention and Response

A SPCC and SPR plan should be evaluated to identify and characterize potential spills, to eliminate or reduce spill potential, and how to respond when a spill occurs. The SPR should include, at a minimum, a copy of the NYSDEC registration application and certificate issued under 6 NYCRR 596.2, a detailed site plan that locates and identifies tanks, transfer stations, and connecting piping, a plan for spill response at the facility which includes a prediction of the flow or dispersion of a spill, a map showing areas that could be impacted by a spill including sewers, wells, ditches, etc., spill reporting procedures, plans for drills, summary of releases occurring in the last five years including reports associated with these releases, identification and assessment of causes of spills, leaks, releases at the Facility, status report on compliance with 6 NYCRR, and the names and phone numbers for emergency contacts, coordinators, and clean-up contractors.

4.3.11 Erosion and Sediment Control

As indicated in Special Condition 5 on page 5 of 9 of the SPDES permit, SWPPPs are not required for discharges of stormwater from construction activity to groundwaters. Since there are no surface waters present at ISP, construction SWPPPs are not required.

4.3.12 Management of Runoff

The BMPs in Appendix D have been proven to be effective at reducing the discharge of pollutants to the stormwater drainage system at ISP.

4.3.13 Street Sweeping

Sweeping can remove small quantities of solids from areas that are exposed to precipitation or stormwater runoff. Sweeping areas that include dust can take place before rainfall or contact with stormwater runoff.

4.4 Activity Specific BMPs

Appendix D identifies recommended BMPs that are applicable to specific airport activities. Each tenant is required to implement at least one (1) BMP for each activity, and the BMP(s) to implement is(are) determined by the tenant and approved by the airport PPT. Appendix D focuses on low-cost source control BMPs and identifies treatment control BMPs such as oil/water separators, where applicable.

These activity specific BMPs are included in Appendix D and apply to the following activities:

BMP 1:	Aircraft Deicing
BMP 2:	Aircraft, Vehicle, and Equipment Fueling
BMP 3:	Aircraft, Vehicle, and Equipment Maintenance
BMP 4:	Aircraft, Vehicle and Equipment Washing, Steam Cleaning and Degreasing

BMP 5:	Building Cleaning and Maintenance
BMP 6:	Chemical and Petroleum Storage and Handling
BMP 7:	Elimination of Non-Storm water Discharges to Storm Drains
BMP 8:	Spills Management
BMP 9:	Oil/Water Separators
BMP 10:	Outdoor Handling of Material
BMP 11:	Outdoor Material and Equipment Storage
BMP 12:	Waste Management
BMP 13:	Stormwater Pollution Prevention Education
BMP 14:	Pavement Sweeping & Stormwater Facility Maintenance
BMP 15:	Security

4.5 Source Reduction

To the maximum extent possible all tenants are required to reduce, reuse, and recycle pollutants generated at ISP. ISP tenants are expected to consider substitute chemicals, segregate activities, and promote recycling. Special Condition - Best Management Practices, Number 6.B.ii (a) and (b) on Page 6 of 9 of the SPDES Permit suggests alternatives to the use of glycol based deicing/anti-icing chemical, evaluate present application rates to ensure against excessive over application by analyzing application rates and adjusting as necessary, consistent with consideration of flight safety, and require that tenants and/or other entities who apply or otherwise use deicing and/or anti-icing materials to determine whether excessive application of deicing/anti-icing chemicals occurs, and adjust as necessary, consistent with consideration of flight safety. The following are some alternatives to the use of glycol-based deicing/anti-icing chemical that are being used at the ISP airport that can reduce the aggregate amount of deicing/anti-icing chemicals used and/or lessen the environmental impact.

4.5.1 Runway Deicing Operations

ISP currently uses sand which is applied to runways , taxiways, and aprons to maintain traction, adjusting as necessary, consistent with flight safety.

4.5.2 Aircraft Deicing/Anti-icing Operations

4.5.2.1 Forced-Air Deicing Systems/Cold Air Blast Deicing

Cold air blast deicing systems use heated compressed air to blow snow and ice off aircraft wings. Air blast deicing may be followed by conventional deicing/anti-icing or accompanied by a fine spray of glycol deicing fluid to prevent new ice formation. A self-contained, truck mounted unit which removes snow and ice from aircraft surfaces by a high-pressure air jet combined with a fine spray of glycol can be used to prevent new ice formation. The airport shall encourage the use of hot air blast deicing systems when tenants replace deicing equipment. ISP used deicing fluids on the runway very infrequently.

4.5.2.2 Optimized Fluid Mixtures

The FAA mixture requirements for "clean aircraft concept" are based on the difference in temperature between the outside air temperature and the freeze point temperature of the deicing mixture. This is known as the "buffer".

For example, a typical 50/50 mixture of a standard Type I deicing fluid and water has a freeze point of -18°F and, therefore, can be used when the outside air temperature is as low as 0°F, allowing for the 18°F buffer.

Airlines typically use a 50/50 mixture of Type I deicing fluid and water for deicing purposes. However, the blend of undiluted deicing fluid and water required to achieve the necessary buffer is dependent on ambient temperature, with the ratio ranging between 60 percent deicing fluid to 40 percent water for temperatures below as 0°F, and 20 percent deicing fluid to 80 percent water for temperatures above -25°F. Hot water alone can be effective at temperatures above 28°F. This fact allows significantly more dilute deicing fluid solutions to remain effective when used at airports located in regions where the temperature rarely falls below 20°F. At the ISP airport some tenants perform regular monitoring of air temperature allowing the use of deicing mixtures with less than the typical 50 percent concentration of glycol, thereby reducing the overall amount of deicing fluid applied. This technique provides. for a direct reduction in the total amount of deicing fluid used with minimal impact on airlines operations. The airport shall encourage the use of such deicing systems when tenants replace deicing equipment.

4.5.2.3 Hybrid Deicing Systems

An innovative aircraft deicing system utilizing forced hot air in combination with a low flow deicing fluid nozzle has been developed. The system is configured similarly to a conventional deicing truck with an operator bucket mounted on a boom and dual tanks for Type I and Type IV deicing and anti-icing fluids. In addition, the unit has a turbine compressor that provides a high velocity air stream, The heart of the system is an applicator turret mounted on the bucket. The applicator head has a ring-shaped nozzle for the air stream and dual (9 and 16 gallons per minute (gpm)) nozzles in the center of the ring for deicing fluid conventional deicing nozzles flow at 45 gpm). Separate controls are provided for each of the nozzles. The applicator head has an operating range of about 10 feet at the 9-gpm flow, and 15-20 feet at 18 gpm. The system can be operated in either deicing or anti-icing modes.

Overall, the hybrid deicing unit can result in a reduction in deicing fluid usage by tenants at ISP airport, Reductions in biological oxygen demand (BOD) loads from glycol can be greater because variable fluid mixtures can be used to suit ambient temperatures, rather than using a single mixture (e.g., 50/50 mix of glycol and water). TOI shall encourage the use of such deicing systems when tenants replace deicing equipment.

4.6 Management of Runoff

Special Condition - Best Management Practices, Number 6.B.iii on Page 6 of 9 of the SPDES Permit indicates that the ISP shall describe and implement a program to control to manage contaminated runoff to reduce the amount of pollutants being discharged at the airport. The stormwater management program shall describe the primary issues that are currently driving decisions regarding the types of deicing runoff for collection systems, recovery systems, and conveyance systems.

To address environmental concerns associated with wintertime aircraft operations, significant effort has been focused on developing solutions for the management of deicing chemical runoff. These included the development of alternative aircraft and runway deicers that are more environmentally friendly, innovative deicing fluid application methods, and alternative collection and treatment methods. ISP developed BMPs to reduce the discharge of deicing and anti-icing materials and manage contaminated runoff to reduce the amount of pollutants being discharged at the airport. Figure 3 in Appendix B indicates the aircraft deicing areas at the passenger terminal plus the drainage system to the recharge basin. The downgradient monitoring wells for propylene glycol

are also indicated in Figure 4. ISP installed a deicing fluid pre-treatment system utilizing a submerged wetlands system as shown on Figure 4 in Appendix B.

5 Comprehensive Site Compliance Evaluation

5.1 BMP Implementation Program

Special Condition - Best Management Practices, Number 6.B.iv and 6.B.v on Page 7 of 9 of the SPDES Permit indicates that TOI shall describe and initiate a BMP implementation program that will specify the routine facility inspections and discuss the comprehensive site compliance evaluations. The implementation program described below is designed to facilitate the proper and timely installation and maintenance of existing and proposed BMPs for ISP tenants.

The BMPs identified in this BMPP were implemented as indicated by NYSDEC to comply with the SPDES Permit. Any changes to the ISP BMPP will be documented and a revised BMPP will be distributed to tenants after revisions are approved by NYSDEC. ISP employees and tenants are provided a copy of the BMPP to review important aspects of the BMPP such as prohibited discharges, inspections, spill response, good housekeeping, and implementation of BMPs, deicing activities, and record keeping procedures.

5.1.1 Site Inspection Report

The results of the Annual Comprehensive Site Compliance Evaluation will document the date, time, inspection methodology, inspectors, BMP conformance evaluation results, and the overall effectiveness of the stormwater pollution prevention program. The results of monitoring efforts conducted throughout the year will also be included with this report.

5.1.1.1 Monthly and Quarterly Outfall Monitoring

The permit requires monthly and quarterly monitoring for the following parameters at Outfalls Number 001 and 002. Monitoring documentation must contain the following information:

- The date, exact place, and time of sampling and measurements;
- The initials or name(s) of the individuals who performed the sampling or measurements;
- The dates) on which analyses were performed;
- The time(s) analyses were initiated;
- The initials or name(s) of the individual(s) who performed the analyses;
- References and written procedures, when available, for the analytical techniques or methods used; and
- The results of laboratory analyses.

Monitoring must be conducted in accordance with test procedures approved under 40 CFR Part 136.

ISP is required to submit the monitoring results in a DMR to the NYSDEC monthly. All monitoring reports, sample data, applications, and other records pertaining to monitoring efforts shall be retained for at least five (5) years from the date of the record.

5.1.1.2 Monthly Deicing/Anti-Icing Season Facility Inspections

The permit requires monthly inspections during deicing/anti-icing season (November through April). If deicing occurs before or after this period, the inspections shall be expanded to include all months during which deicing chemical may be used. Also, if a significant or deleteriously large quantities of deicing chemical are spills or discharged, or if water quality impacts have been reported, the inspection frequency shall be increased to weekly until such time as the chemical spills/discharge or impacts are reduced to acceptable levels.

Tenants who conduct deicing/anti-icing operations must provide the TOI BMPP Coordinator a monthly deicing/anti-icing usage. Tenants and ISP maintain a record of the types and monthly quantities of deicing/anti-icing chemical used (including the Material Safety Data Sheets (MSDSs).

TOI conducts monthly inspections of deicing/anti-icing materials storage and handling areas from November through April.

5.1.1.3 BMPP Implementation Inspections

An Annual Site Compliance Evaluation included in Appendix E includes procedures and protocols for inspections to be performed to adhere to specific BMPs identified in the BMPP required by the SPDES Permit. TOI personnel and tenants operating at ISP perform self-inspections of material storage areas, material handling areas, and disposal areas to assess and make sure their facilities comply with the BMPs. Tenants are responsible for addressing all non-compliance issues at their respective facilities in accordance with Federal, State, and local environmental regulations as per their lease agreements. An Annual Site Compliance Evaluation will be used to assess BMPP implementation effectiveness.

5.1.2 Discharge and Spill Reporting

Any results of outfall monitoring required above, excluding screening data, must be submitted to the NYSDEC by appending them to the corresponding DMR. Failure to perform the required discharge monitoring and reporting shall constitute a violation of the terms of the SPDES permit.

Tenants are required to notify TOI and Fire Rescue with a spill of any size identifying the event date/time, location, type of spill, response, gallons spilled, cause/reason for spill or leak, and recommended remedial action.

The Fire Rescue team will respond to the incident to ensure the spill is being handled correctly. In the event the air carrier cannot handle the release TOI will mitigate the spill with the staff of ISP Fire Rescue and Maintenance.

If ISP Fire Rescue and Maintenance are unable to handle the spill, TOI contacts the TOI Haz-Mat Response Team and if further assistance is needed TOI will request assistance from the Suffolk County Strike Team.

5.1.3 BMPP Content Review

BMPP elements will be reviewed annually. Any necessary revisions to the BMPP, based on the facility inspections, will be documented, and incorporated. The BMPP will also be amended if there are changes in construction, operation, or maintenance that may affect the discharge of pollutants to surface water, groundwater, or the stormwater drainage system. Individual tenants are required to notify the ISP BMPP coordinator or their representative as early as feasible when contemplating any such changes. The BMPP will also be modified if certain BMPs are shown to be ineffective in achieving the general objective of controlling pollutants in stormwater.

An Annual Site Compliance Evaluation included in Appendix E was an addition to the BMPP to assist TOI and tenants with review of procedures and protocols for inspections to be performed to adhere to specific BMPs identified in the BMPP required by the SPDES Permit. The Annual Site Compliance Evaluation will be used to assess BMPP implementation effectiveness.

5.1.4 Comprehensive Site Compliance Evaluation

An inspection of tenant facilities will be conducted, and the frequency of the inspections is as follows:

- Annual inspections by PPT personnel (accompanied by the designated tenant representative) to verify that all BMPP elements are properly implemented at the facility.
- Routine self-inspection by tenants of their leaseholds/operations to ensure that BMPs have been and continue to be properly implemented.
- Deicing operations inspections will be scheduled during the deicing season.
- If the inspection is not practical during active deicing or the weather is inclement, the inspection will be conducted when operations are likely to occur, and the material and equipment of deicing are in place.
- Deicing samples shall be conducted once per month during deicing/anti-icing season (November to April).
- Deicing inspections shall be expanded to include all the months during which deicing chemicals may be used.
- Deicing inspections will be conducted if significantly or deleteriously large quantities of deicing chemicals are being spilled or discharged, or if water quality impacts have been reported, the inspection frequency shall be increased to weekly until such time as the chemical spills/discharges or impacts are reduced to acceptable levels.

The Annual Site Compliance Evaluation should include visual inspections of activities potentially impacting stormwater, the need for additional BMPs, and evidence of pollutants entering the drainage system. A report documenting the scope, observations, and any instances of non-compliance and recommendations of the Annual Site Compliance Evaluation should be added to the BMPP and retained as part of the BMPP until one year after the expiration of the permit.

The PPT personnel designated and trained to implement the BMPP will perform the Annual Site Compliance Evaluation and provide the ISP BMPP coordinator or their representative with complete and accurate information. All inspections will be carefully documented and required changes will be incorporated into the BMPP. These records will be retained until three (3) years after the coverage from the current industrial permit is terminated.

Appendix A

SPDES Permit for ISP

New York State Department of Environmental Conservation
Division of Environmental Permits
NYSDEC HEADQUARTERS
625 BROADWAY
ALBANY, NY 12233
(518) 402-9167

SPDES PERMIT RENEWAL

12/19/2017

ROBERT A SCHNEIDER
LONG ISLAND MACARTHUR AIRPORT
100 ARRIVAL AVE
RONKONKOMA NY 11779

Permittee Name: TOWN OF ISLIP
Facility Name: LONG ISLAND MACARTHUR AIRPORT
Ind. Code: 4581 County: SUFFOLK
DEC ID: 1-4728-00783/00003 SPDES No.: NY0173703
Permit Effective Date: 9/1/2018
Permit Expiration Date: 8/31/2028

Dear Permittee,

The State Pollutant Elimination System (SPDES) permit renewal for the facility referenced above is approved with the new effective and expiration dates. This letter together with the previous valid permit for this facility effective on 09/01/2013 and any subsequent modifications constitute authorization to discharge wastewater in accordance with all terms, conditions and limitations specified in the previously issued permit(s).

As a reminder, SPDES permits are renewed at a central location in Albany in order to make the process more efficient. All other concerns with your permit, including applications for permit modification or transfer to a new owner, a name change, and other questions, should be directed to:

Regional Permit Administrator
NYSDEC Region 1 Headquarters
SUNY @ Stony Brook|50 Circle Rd
Stony Brook, NY 11790-3409
(631) 444-0365

If you have already filed an application for modification of your permit, it will be processed separately by that office.

If you have questions concerning this permit renewal, please contact MICHAEL R SCHAEFER at (518) 402-9167.

Sincerely,

Kent P. Sanders
Deputy Permit Administrator

cc:
RPA
BWC
RWE
File
BWP

New York State Department of Environmental Conservation
Division of Environmental Permits, Region One
SUNY @ Stony Brook, 50 Circle Road, Stony Brook, NY 11790-3409
Phone: (631) 444-0365 **Fax:** (631) 444-0360
Website: www.dec.ny.gov

Joe Martens
Commissioner

May 17, 2013

Long Island MacArthur Airport
Attn: Robert A. Schneider
100 Arrival Avenue
Ronkonkoma NY 11779

Re: Permit # 1-4728-00783/00003
SPDES Number: NY0173703

Dear Permittee:

In conformance with the requirements of the State Uniform Procedures Act (Article 70, ECL) and its implementing regulations (6 NYCRR, Part 621) we are enclosing your permit. Please carefully read all permit conditions and special permit conditions contained in the permit to ensure compliance during the term of the permit. If you are unable to comply with any conditions, please contact us at the above address.

Sincerely,

Matthew R. Penski
Environmental Analyst 1

cc: CO BWP – Permit Coordinator
Water, R1
File

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

State Pollutant Discharge Elimination System (SPDES)
DISCHARGE PERMIT

First3.99

Industrial Code:	**4581**	SPDES Number:	**NY0173703**
Discharge Class (CL):	**01**	DEC Number:	**1-4728-00783/00003**
Toxic Class (TX):	**T**	Effective Date (EDP):	**September 1, 2008**
Major Drainage Basin:	**17**	Expiration Date (ExDP):	**August 31, 2013**
Sub Drainage Basin:	**01**	Modification Dates: (EDPM)	**May 31, 2013**
Water Index Number:	**Groundwater**		
Compact Area:			

This SPDES permit is issued in compliance with Title 8 of Article 17 of the Environmental Conservation Law of New York State and in compliance with the Clean Water Act, as amended, (33 U.S.C. §1251 et.seq.)(hereinafter referred to as "the Act").

PERMITTEE NAME AND ADDRESS

Name:	**Long Island MacArthur Airport**	Attention:	**Robert A. Schneider**		
Street:	**100 Arrival Avenue**				
City:	**Ronkonkoma**	State:	**NY**	Zip Code:	**11779**

is authorized to discharge from the facility described below:

FACILITY NAME AND ADDRESS

Name:	Long Island MacArthur Airport										
Location (C,T,V):	**(T) Islip**						County:	**Suffolk**			
Facility Address:	100 Arrival Avenue										
City:	**Ronkonkoma**				State:	**NY**		Zip Code:	**11779**		
NYTM -E:					NYTM - N:						
From Outfall No.:	**001**	at Latitude:	**40** °	**47** ‘	**23** “	& Longitude:	**73** °	**06** ‘	**23**		
into receiving waters known as:	**Groundwater**						Class:	**GA**			

and (list other Outfalls, Receiving Waters & Water Classifications)
002, 003 Groundwater – GA

in accordance with: effluent limitations; monitoring and reporting requirements; other provisions and conditions set forth in this permit; and 6 NYCRR Part 750-1and 750-2.

DISCHARGE MONITORING REPORT (DMR) MAILING ADDRESS

Mailing Name:	**Robert A. Schneider – Long Island MacArthur Airport**				
Street:	**100 Arrival Avenue**				
City:	**Ronkonkoma**	State:	**NY**	Zip Code:	**11779**
Responsible Official or Agent:	**Robert A. Schneider**			Phone:	**631-467-3300**

This permit and the authorization to discharge shall expire on midnight of the expiration date shown above and the permittee shall no discharge after the expiration date unless this permit has been renewed, or extended pursuant to law. To be authorized to discharge beyond th expiration date, the permittee shall apply for permit renewal not less than 180 days prior to the expiration date shown above.

DISTRIBUTION:

CO BWP - Permit Coordinator
RWE
RPA

Permit Administrator: John A. Wieland, Deputy Permit Administrator

Address: 50 Circle Road
Stony Brook, NY 11790-3409

Signature:

Date: 05/17/20

PERMIT LIMITS, LEVELS AND MONITORING DEFINITIONS

OUTFALL	WASTEWATER TYPE	RECEIVING WATER	EFFECTIVE	EXPIRING
	This cell describes the type of wastewater authorized for discharge. Examples include process or sanitary wastewater, storm water, non-contact cooling water.	This cell lists classified waters of the state to which the listed outfall discharges.	The date this page starts in effect. (e.g. EDP or EDPM)	The date this page is no longer in effect. (e.g. ExDP)

PARAMETER	MINIMUM	MAXIMUM	UNITS	SAMPLE FREQ.	SAMPLE TYPE
e.g. pH, TRC, Temperature, D.O.	The minimum level that must be maintained at all instants in time.	The maximum level that may not be exceeded at any instant in time.	SU, °F, mg/l, etc.	See below	See below

PARAMETER	EFFLUENT LIMIT or CALCULATED LEVEL	COMPLIANCE LEVEL/ ML	ACTION LEVEL	UNITS	SAMPLE FREQUENCY	SAMPLE TYPE
	Limit types are defined below in Note 1. The effluent limit is developed based on the more stringent of technology-based limits, required under the Clean Water Act, or New York State water quality standards. The limit has been derived based on existing assumptions and rules. These assumptions include receiving water hardness, pH and temperature; rates of this and other discharges to the receiving stream; etc. If assumptions or rules change the limit may, after due process and modification of this permit, change.	For the purposes of compliance assessment, the permittee shall use the approved EPA analytical method with the lowest possible detection limit as promulgated under 40CFR Part 136 for the determination of the concentrations of parameters present in the sample unless otherwise specified. If a sample result is below the detection limit of the most sensitive method, compliance with the permit limit for that parameter was achieved. Monitoring results that are lower than this level must be reported, but shall not be used to determine compliance with the calculated limit. This PQL can be neither lowered nor raised without a modification of this permit.	Action Levels are monitoring requirements, as defined below in Note 2, which trigger additional monitoring and permit review when exceeded.	This can include units of flow, pH, mass, temperature, or concentration . Examples include µg/l, lbs/d, etc.	Examples include Daily, 3/week, weekly, 2/month, monthly, quarterly, 2/yr and yearly. All monitoring periods (quarterly, semiannual, annual, etc) are based upon the calendar year unless otherwise specified in this Permit.	Examples include grab, 24 hour composite and 3 grab samples collected over a 6 hour period.

Notes:

1. EFFLUENT LIMIT TYPES:
   a. DAILY DISCHARGE: The discharge of a pollutant measured during a calendar day or any 24-hour period that reasonably represents the calendar da for the purposes of sampling. For pollutants expressed in units of mass, the 'daily discharge' is calculated as the total mass of the pollutant discharge over the day. For pollutants with limitations expressed in other units of measurement, the 'daily discharge' is calculated as the average measurement the pollutant over the day.
   b. DAILY MAX.: The highest allowable daily discharge. DAILY MIN.: The lowest allowable daily discharge.
   c. MONTHLY AVG: The highest allowable average of daily discharges over a calendar month, calculated as the sum of each of the daily discharge measured during a calendar month divided by the number of daily discharges measured during that month.
   d. 7 DAY ARITHMETIC MEAN (7 day average): The highest allowable average of daily discharges over a calendar week.
   e. 30 DAY GEOMETRIC MEAN: The highest allowable geometric mean of daily discharges over a calendar month, calculated as the antilog of: the sum the log of each of the daily discharges measured during a calendar month divided by the number of daily discharges measured during that month.
   f. 7 DAY GEOMETRIC MEAN: The highest allowable geometric mean of daily discharges over a calendar week.
   g. RANGE: The minimum and maximum instantaneous measurements for the reporting period must remain between the two values shown.

2. ACTION LEVELS: Routine Action Level monitoring results, if not provided for on the Discharge Monitoring Report (DMR) form, shall be appended to th DMR for the period during which the sampling was conducted. If the additional monitoring requirement is triggered as noted below, the permittee sha undertake a short-term, high-intensity monitoring program for the parameter(s). Samples identical to those required for routine monitoring purposes shall b taken on each of at least three consecutive operating and discharging days and analyzed. Results shall be expressed in terms of both concentration and mas and shall be submitted no later than the end of the third month following the month when the additional monitoring requirement was triggered. Results may b appended to the DMR or transmitted under separate cover to the same address. If levels higher than the Action Levels are confirmed, the permit may b reopened by the Department for consideration of revised Action Levels or effluent limits. The permittee is not authorized to discharge any of the liste parameters at levels which may cause or contribute to a violation of water quality standards.

PERMIT LIMITS, LEVELS AND MONITORING

OUTFALL	WASTEWATER TYPE	RECEIVING WATER	EFFECTIVE	EXPIRING
001	Stormwater from Tank Farm Area	Groundwater	EDPM	ExDP

PARAMETER	MINIMUM	MAXIMUM	UNITS	SAMPLE FREQUENCY	SAMPLE TYPE	FOOTNOTES (FN)
pH	5.5	8.5	SU	Monthly	Grab	

PARAMETER	EFFLUENT LIMIT or CALCULATED LEVEL		COMPLIANCE LEVEL/ ML	ACTION LEVEL	UNITS	SAMPLE FREQUENCY	SAMPLE TYPE	FN
	Monthly Avg	Daily Max						
Oil & Grease	NA	15			mg/L	Monthly	Grab	
Benzene	NA	0.7			ug/L	Monthly	Grab	
Toluene	NA	5			ug/L	Monthly	Grab	
Xylene	NA	5			ug/L	Monthly	Grab	1
Ethylbenzene	NA	5			ug/L	Monthly	Grab	

OUTFALL	WASTEWATER TYPE	RECEIVING WATER	EFFECTIVE	EXPIRING
002	Stormwater from Ramp Area	Groundwater	EDPM	ExDP

PARAMETER	MINIMUM	MAXIMUM	UNITS	SAMPLE FREQUENCY	SAMPLE TYPE	FOOTNOTES (FN)
pH	5.5	8.5	SU	Monthly	Grab	

PARAMETER	EFFLUENT LIMIT or CALCULATED LEVEL		COMPLIANCE LEVEL/ ML	ACTION LEVEL	UNITS	SAMPLE FREQUENCY	SAMPLE TYPE	FN
	Monthly Avg	Daily Max						
Oil & Grease	NA	15			mg/L	Monthly	Grab	
Benzene	NA	0.7			ug/L	Monthly	Grab	
Toluene	NA	5			ug/L	Monthly	Grab	
Xylene	NA	5			ug/L	Monthly	Grab	1
Ethylbenzene	NA	5			ug/L	Monthly	Grab	
Total Nitrogen	NA	10			mg/L	Monthly	Grab	2
Total Phosphorus	NA	Monitor			mg/L	Monthly	Grab	2
Propylene Glycol	NA	1			mg/L	Monthly	Grab	3

OUTFALL	WASTEWATER TYPE	RECEIVING WATER	EFFECTIVE	EXPIRING
003	Sanitary Discharges – No Monitoring Required	Groundwater	EDPM	ExDP

FOOTNOTES:

1. Applicable to each isomer: m, o, and p individually.
2. Samples are to be collected monthly from November to April.
3. Propylene Glycol samples are to be collected from downgradient monitoring wells MW1 & MW2. These samples are to be collected monthly from November to April.

SPECIAL CONDITIONS - INDUSTRY BEST MANAGEMENT PRACTICES

1. **General** - The permittee shall develop, maintain, and implement a Best Management Practices (BMP) plan to prevent releases of significant amounts of pollutants to the waters of the State through plant site runoff; spillage and leaks; sludge or waste disposal; and stormwater discharges including, but not limited to, drainage from raw material storage. The BMP plan shall be documented in narrative form and shall include the 13 minimum BMPs and any necessary plot plans, drawings, or maps. Other documents already prepared for th facility such as a Safety Manual or a Spill Prevention, Control and Countermeasure (SPCC) plan may be used as part of the plan and may be incorporated by reference. A copy of the current BMP plan shall be submitted to the Department as required in item (2.) below and a copy must be maintained at the facility and shall be available to authorized Department representatives upon request.

2. **Compliance Deadlines** - A completed BMP plan was submitted January 28, 2010. The BMP plan shall be reviewed annually and shall b modified whenever (a) changes at the facility materially increase the potential for releases of pollutants; (b) actual releases indicate the plan is inadequate, or (c) a letter from the Department identifies inadequacies in the plan. The permittee shall certify in writing, as an attachment to the December Discharge Monitoring Report (DMR), that the annual review has been completed. All BMP plan revisions (with the exception of SWPPPs - see item (5.) below) must be submitted to the Regional Water Engineer within 30 days. Note that the permittee is not required to obtain Department approval of the BMP plan (or of any SWPPPs) unless notified otherwise. Subsequent modifications to or renewal of this permit does not reset or revise these deadlines unless a new deadline is set explicitly by such permit modification or renewal.

3. **Facility Review** - The permittee shall review all facility components or systems (including but not limited to material storage areas; in-plant transfer, process, and material handling areas; loading and unloading operations; storm water, erosion, and sediment control measures; process emergency control systems; and sludge and waste disposal areas) where materials or pollutants are used, manufactured stored or handled to evaluate the potential for the release of pollutants to the waters of the State. In performing such an evaluation, the permittee shall consider such factors as the probability of equipment failure or improper operation, cross-contamination of storm water b process materials, settlement of facility air emissions, the effects of natural phenomena such as freezing temperatures and precipitation, fires, and the facility's history of spills and leaks. The relative toxicity of the pollutant shall be considered in determining the significance of potential releases. The review shall address all substances present at the facility that are identified in Tables 6-10 of SPDES applicatio Form NY-2C (available at http://www.dec.ny.gov/docs/permits_ej_operations_pdf/form2c.pdf) or that are required to be monitored for t the SPDES permit.

4. **13 Minimum BMPs:** Whenever the potential for a release of pollutants to State waters is determined to be present, the permittee shall identify BMPs that have been established to prevent or minimize such potential releases. Where BMPs are inadequate or absent, appropriate BMPs shall be established. In selecting appropriate BMPs, the permittee shall consider good industry practices and, where appropriate, structural measures such as secondary containment and erosion/sediment control devices and practices. USEPA guidance fo development of stormwater elements of the BMP is available in the September 1992 manual *Storm Water Management for Industrial Activities,* EPA 832-R-92-006 (available from NTIS, 703-487-4650, order # PB 92235969). As a minimum, the plan shall include the following BMPs:

    1. BMP Pollution Prevention Team
    2. Reporting of BMP Incidents
    3. Risk Identification & Assessment
    4. Employee Training
    5. Inspections and Records
    6. Security
    7. Preventive Maintenance
    8. Good Housekeeping
    9. Materials/Waste Handling, Storage, & Compatibility
    10. Spill Prevention & Response
    11. Erosion & Sediment Control
    12. Management of Runoff
    13. Street Sweeping

    Note that for some facilities, especially those with few employees, some of the above BMPs may not be applicable. It is acceptable in these cases to indicate "Not Applicable" for the portion(s) of the BMP Plan that do not apply to your facility, along with an explanation.

SPECIAL CONDITIONS - INDUSTRY BEST MANAGEMENT PRACTICES (continued)

5. **Stormwater Pollution Prevention Plans (SWPPPs) Required for Discharges of Stormwater From Construction Activity to Surfac Waters** - As part of BMP #11, a SWPPP shall be developed prior to the initiation of any site disturbance of one acre or more of uncontaminated area. Uncontaminated area means soils or groundwater which are free of contamination by any toxic or non-conventiona pollutants identified in Tables 6-10 of SPDES application Form NY-2C. Disturbance of any size contaminated area(s) and the resulting discharge of contaminated stormwater is not authorized by this permit unless the discharge is under State or Federal oversight as part of a remedial program or after review by the Regional Water Engineer; nor is such discharge authorized by any SPDES general permit for stormwater discharges. SWPPPs are not required for discharges of stormwater from construction activity to groundwaters. The SWPPP shall conform to the *New York Standards and Specifications for Erosion and Sediment Control* and *New York State Stormwater Management Design Manual,* unless a variance has been obtained from the Regional Water Engineer, and to any local requirements. The permittee shall submit a copy of the SWPPP and any amendments thereto to the local governing body and any other authorized agency having jurisdiction or regulatory control over the construction activity **at least 30 days prior to soil disturbance**. The SWPPP shall also be submitted to the Regional Water Engineer if contamination, as defined above, is involved and the permittee must obtain a determination of any SPDES permit modifications and/or additional treatment which may be required prior to soil disturbance. Otherwise the SWPPP shall be submitted to the Department only upon request. When a SWPPP is required, a properly completed *Notice of Intent* (NOI) form shall be submitted (available at www.dec.ny.gov/chemical/43133.html) prior to soil disturbance. Note that submission of a NOI is required for informational purposes; the permittee is not eligible for and will not obtain coverage under any SPDES general permi for stormwater discharges, nor are any additional permit fees incurred. SWPPPs must be developed and submitted for subsequent site disturbances in accordance with the above requirements. The permittee is responsible for ensuring that the provisions of each SWPPP ar properly implemented.\

6. **Airports:** The BMP plan must include pertinent elements of the SWPPP for industrial activities.
The requirements listed under this section apply to stormwater discharges associated with industrial activity from air transportation facilities including air transportation (scheduled and non-scheduled); air courier services; airports; flying fields (except those maintained by aviation clubs); air terminal services including air traffic control (except government); aircraft storage at airports; aircraft upholstery repair; airfreight handling at airports; airport hangar rental; airport leasing, if operating airport; airport terminal services; hangar operation airport, aircraft service and maintenance including aircraft cleaning and janitorial service; aircraft servicing /repairing except on a factory basis); vehicle maintenance shops; material handling facilities; equipment clearing operations; and airport/aircraft deicing and anti-icing. [Note: For the purpose of this section, the term "deicing" is defined as the process to remove frost, snow, or ice and "anti-icing" is the process which prevents the accumulation of frost, snow, or ice.] Only those portions of the facility that are either involved in vehicle maintenance (including vehicle rehabilitation, mechanical repairs, painting, fueling, and lubrication), equipment cleaning operations, or deicing/anti-icing operations are addressed under this section. Tenants and/or other entities that apply or otherwise use deicing and/or anti-icing materials shall provide all necessary information to the permittee for the permittee to complete all requirements under this section.

Additional Requirements for the BMP Plan: BMPs shall be developed for areas of the facility occupied by tenants of the airport and shall be integrated with the BMP plan for the entire airport. For the purposes of this permit, tenants of the airport facility include airline passenger or cargo companies, fixed based operators and other parties who have contracts with the airport authority to conduct business operations on airport property and whose operations result in stormwater discharges associated with industrial activity. The BMP plan shall include, at a minimum, the following items.

A. Site description

(i) Site map - The site map shall identify where any of the following activities may be exposed to precipitation/surface runoff: aircraf and runway deicing/anti-icing operations; fueling stations; aircraft, ground vehicle and equipment maintenance/cleaning areas; and storage areas for aircraft, ground vehicles and equipment awaiting maintenance.

(ii) Summary of potential pollutant sources - A narrative description of the potential pollutant sources from the following activities: aircraft, runway, ground vehicle and equipment maintenance and cleaning; aircraft and runway deicing/anti-icing operations (including apron and centralized aircraft deicing/anti-icing stations, runways, taxiways and ramps). Facilities which conduct deicing/anti-icing operations shall maintain a record of the types (including the Material Safety Data Sheets (MSDS)) and monthly quantities of deicing/anti-icing chemicals used, either as measured amounts, or in the absence of metering, as estimated amounts. This includes all deicing/anti-icing chemicals, not just glycols and urea (e.g., potassium acetate). Tenants and fixed-base operators who conduct deicing/anti-icing operations shall provide the above information to the airport authority for inclusion in the BMP for th entire facility.

B. Stormwater controls

(i) Good housekeeping

(a) Aircraft, ground vehicle and equipment maintenance areas - The permittee must describe and implement measures that prevent or minimize the contamination of stormwater runoff from all areas used for aircraft, ground vehicle and equipment maintenance (including the maintenance conducted on the terminal apron and in dedicated hangars). The following practices (or their equivalents) shall be considered: performing maintenance activities indoors; maintaining an organized inventory of materials used in the maintenance areas; draining all parts of fluids prior to disposal; preventing the practice of hosing down the apron or hangar floor; using dry cleanup methods ; and collecting the stormwater runoff from the maintenance area and providin treatment or recycling.

(b) Aircraft, ground vehicle and equipment cleaning areas - Permittees shall ensure that cleaning of equipment is conducted in designated areas only and clearly identify these areas on the ground and delineate them on the site map. The permittee must describe and implement measures that prevent or minimize the contamination of the stormwater runoff from cleaning areas.

(c). Aircraft, ground vehicle and equipment storage areas - The storage of aircraft, ground vehicles and equipment awaiting maintenance must be confined to designated areas (delineated on the site map). The following BMPs (or their equivalents) shall be considered: indoor storage of aircraft and ground vehicles; the use of drip pans for the collection of fluid leaks; and perimeter drains, dikes or berms surrounding storage areas.

(d) Material storage areas - Storage vessels of all materials (e.g., used oils, hydraulic fluids, spent solvents, and waste aircraft fuel) must be maintained in good condition, so as to prevent or minimize contamination of stormwater, and plainly labeled (e.g., "used oil," "Contaminated Jet A," etc.). The permittee must describe and implement measures that prevent or minimize contamination of precipitation/runoff from storage areas. The following BMPs or their equivalents shall be considered: indoor storage of materials centralized storage areas for waste materials; and installation of berms/dikes around storage areas.

(e) Airport fuel system and fueling areas - The permittee must describe and implement measures that prevent or minimize the discharge of fuels to the storm sewer/surface waters resulting from fuel servicing activities or other operations conducted in support of the airport fuel system. The following BMPs (or their equivalents) shall be considered: implementing spill and overflow practices (e.g., placing absorptive materials beneath aircraft during fueling operations); using dry cleanup methods; and collecting the stormwater runoff.

(ii) Source reduction - The permittee shall consider alternatives to the use of urea and glycol-based airfield deicing/anti-icing chemicals to reduce the aggregate amount of airfield deicing/anti-icing. The permittee shall require the tenants and/or other entities who apply or otherwise use deicing and/or anti-icing materials to consider alternatives to the use of urea and glycol-based deicing/anti-icing chemicals to reduce the aggregate amount of deicing/anti-icing chemicals used and/or lessen the environmental impact. Chemical options to replace ethylene glycol, propylene glycol and urea include: potassium acetate; magnesium acetate; calcium acetate; anhydrous potassium acetate.

(a) Runway deicing operations - The Permittee shall evaluate present application rates to ensure against excessive over application by analyzing application rates and adjusting as necessary, consistent with considerations of flight safety. Also the following BMP options shall be considered (or their equivalents): metered application of chemicals; prewetting dry chemical constituents prior to application; installation of runway ice detection systems; implementing anti-icing operations as a preventive measure against ice buildup.

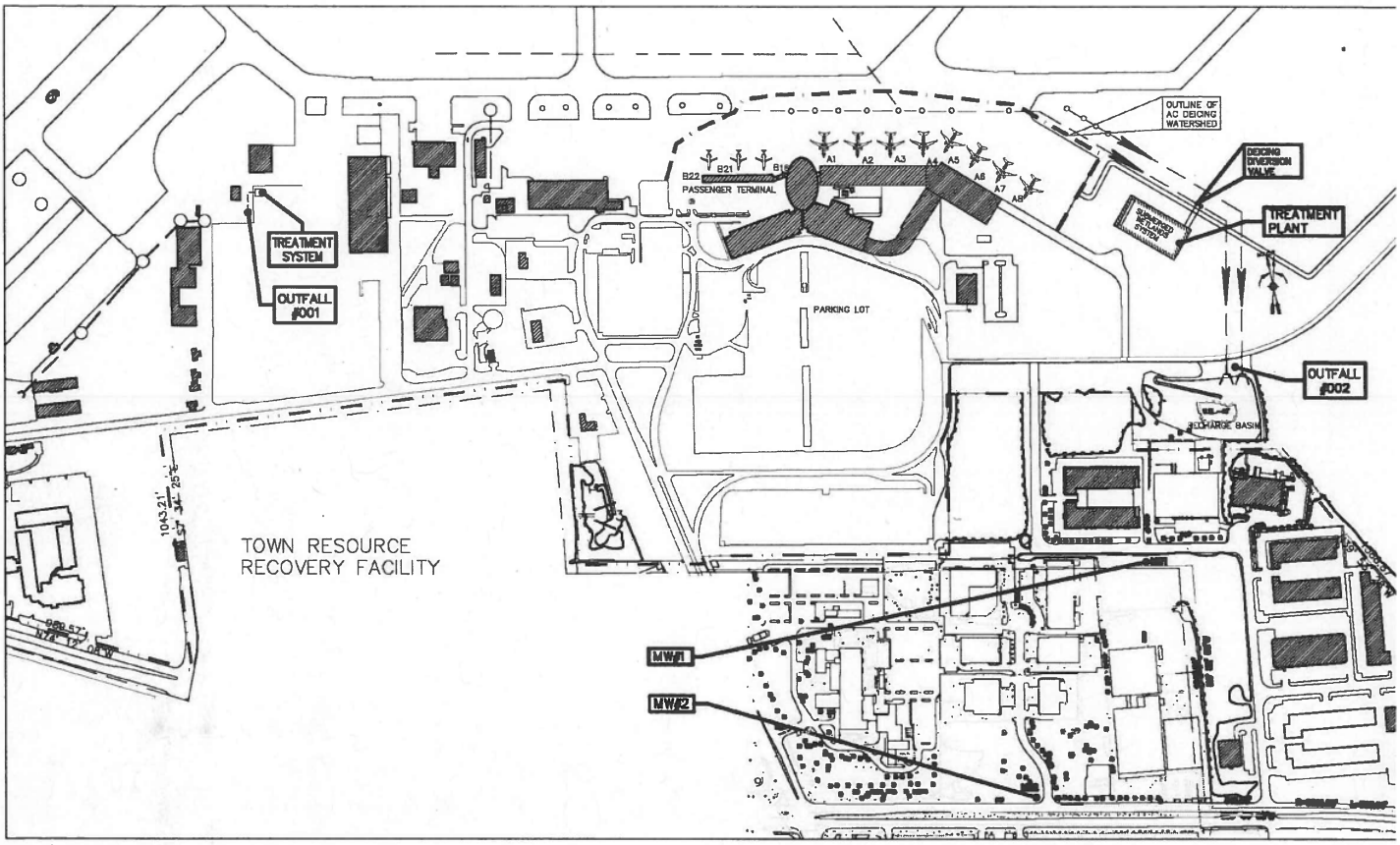
(b) Aircraft deicing/anti-icing operations - The Permittee shall require tenants and/or other entities who apply or otherwise use deicing and/or anti-icing materials to determine whether excessive application of deicing/anti-icing chemicals occurs, and adjust as necessary, consistent with considerations of flight safety. This evaluation should be carried out by the personnel most familia with the particular aircraft and flight operations in question (versus an outside entity such as the airport authority). The use of alternative deicing/anti-icing agents as well as containment measures for all applied chemicals shall be considered. Also, the following BMP options (or their equivalents) shall be considered for reducing deicing fluid use: forced-air deicing systems; computer-controlled fixed-gantry systems; infrared technology; hot water; varying glycol content to air temperature; enclosed-basket deicing trucks; mechanical methods; solar radiation; hangar storage; aircraft covers; and thermal blankets for MD-80s and DC-9s. The use of ice-detection systems and airport traffic flow strategies and departure slot allocation systems shall also be considered.

(iii) Management of runoff - Where deicing/anti-icing operations occur, the permittee, tenants and/or other entities who apply or otherwise use deicing and/or anti-icing materials shall describe and implement a program to control or manage contaminated runoff t reduce the amount of pollutants being discharged from the site. The following BMPs (or their equivalents) shall be considered: establishing a dedicated deicing facility with a runoff collection/recovery system; using vacuum/collection trucks; storing contaminated stormwater/deicing fluids in tanks and releasing controlled amounts to a publicly owned treatment works; collecting contaminated runoff in a wet pond for biochemical decomposition (be aware of attracting wildlife that may prove hazardous to flight operations); and directing runoff into vegetative swales or other infiltration measures. The plan shall consider the recovery of deicing/anti-icing materials when these materials are applied during nonprecipitation events (e.g., covering storm sewer inlets, using booms, installing absorptive interceptors in the drains, etc.) tc prevent these materials from later becoming a source of stormwater contamination. Used deicing fluid should be recycled whenever possible.

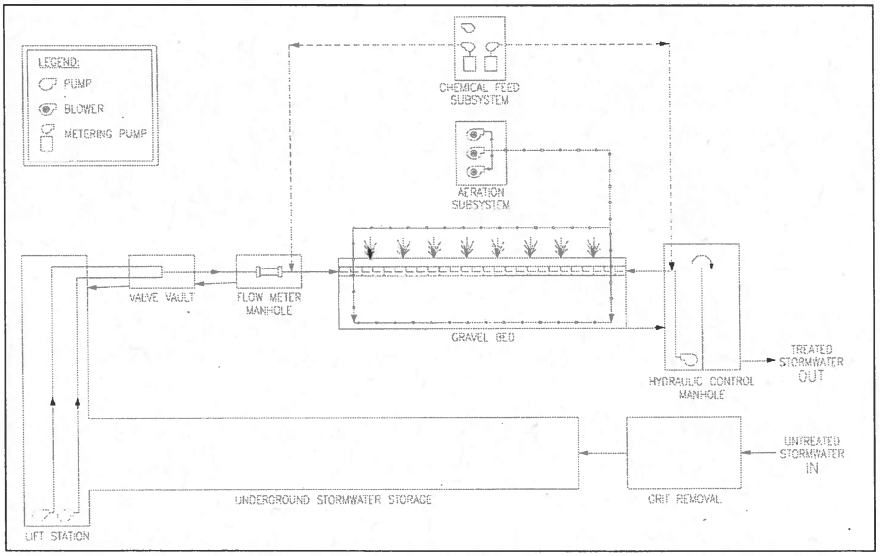
(iv) Routine facility inspections - The inspection frequency shall be specified in the plan. At a minimum, inspections shall be conducted once per month during deicing/anti-icing season (e.g., October through April for most airports). If deicing occurs before or after this period, the inspections shall be expanded to include all months during which deicing chemicals may be used. Also, if significantly or deleteriously large quantities of deicing chemicals are being spilled or discharged, or if water quality impacts have been reported, the inspection frequency shall be increased to weekly until such time as the chemical spills/discharges or impacts are reduced to acceptable levels.

(v) Comprehensive site compliance evaluation - The annual site compliance evaluations shall be conducted by qualified facility personnel during periods of actual deicing operations, if possible. If not practicable during active deicing or if the weather is too inclement, the evaluations shall be conducted when deicing operations are likely to occur and the materials and equipment for deicin are in place.

MONITORING LOCATIONS



Facility Map



Water flow diagram for Outfall 002

RECORDING, REPORTING AND ADDITIONAL MONITORING REQUIREMENTS

a) 6 NYCRR Part 750 is hereby incorporated by reference and its conditions are enforceable requirements of this permit. The permittee sha comply with all conditions set forth in this permit and with 6 NYCRR Part 750, including, but not limited to: additional monitoring an reporting requirements and conditions, including noncompliance reporting.

b) The monitoring information required by this permit shall be summarized, signed and retained for a period of at least five years from the date the sampling for subsequent inspection by the Department or its designated agent. **Also, monitoring information required by this perm shall be summarized and reported by submitting;**

[X] (if box is checked) completed and signed Discharge Monitoring Report (DMR) forms for each _1_ month reporting period to the locations specified below. Blank forms are available at the Department's Albany office listed below. The first reporting period begins on the effective date of this permit and the reports will be due no later than the 28th day of the month following the end of each reporting period.

[ ] (if box is checked) an annual report to the Regional Water Engineer at the address specified below. The annual report is due by February 1 each year and must summarize information for January to December of the previous year in a format acceptable to the Department.

[ ] (if box is checked) a monthly "Wastewater Facility Operation Report..." (form 92-15-7) to the:
[ ] Regional Water Engineer and/or [ ] County Health Department or Environmental Control Agency specified below

Send the **original** of each DMR page to:
Department of Environmental Conservation
Division of Water
Bureau of Water Compliance
625 Broadway, Albany, NY 12233-3506
Phone: (518) 402-8177

Send a **copy** of each DMR page to:
Department of Environmental Conservation
Regional Water Engineer
SUNY @ Stony Brook
50 Circle Road
Stony Brook, NY 11790-3409
Phone: 631-444-0405

c) Monitoring and analysis shall be conducted according to test procedures approved under 40 CFR Part 136, unless other test procedure have been specified in this permit.

d) More frequent monitoring of the discharge(s), monitoring point(s), or waters of the State than required by the permit, where analysis performed by a certified laboratory or where such analysis is not required to be performed by a certified laboratory, shall be included in th calculations and recording of the data on the corresponding DMRs.

e) Calculations which require averaging of measurements shall utilize an arithmetic mean unless otherwise specified in this permit.

f) Unless otherwise specified, all information recorded on the DMRs shall be based upon measurements and sampling carried out during th most recently completed reporting period.

g) Any laboratory test or sample analysis required by this permit for which the State Commissioner of Health issues certificates of approv pursuant to section 502 of the Public Health Law shall be conducted by a laboratory which has been issued a certificate of approva Inquiries regarding laboratory certification should be directed to the New York State Department of Health, Environmental Laborato Accreditation Program.

New York State Department of Environmental Conservation
Division of Environmental Permits, Region One
SUNY @ Stony Brook, 50 Circle Road, Stony Brook, NY 11790 - 3409
Phone: (631) 444-0365 • **FAX:** (631) 444-0360
Website: www.dec.state.ny.us

Alexander B. Grannis
Commissioner

PERMIT MODIFICATION

September 22, 2009

Long Island Macarthur Airport
Attn: Robert A. Schneider
100 Arrival Avenue
Ronkonkoma NY 11779

Re: Permit # 1-4728-00783/00003
Long Island Macarthur Airport
SPDES # NY0173703

Dear Permittee:

The Department of Environmental Conservation has reviewed your request to modify the above permit and finds the changes to be acceptable. Therefore, in conformance with the requirements of the State Uniform Procedures Act (Article 70, ECL) and its implementing regulations (6NYCRR, Part 621) we are enclosing your **modified** permit. Please carefully read all permit conditions and special permit conditions contained in the permit to insure compliance during the term of the permit. If you are unable to comply with any conditions contact us at the above address.

This permit amendment must be kept available on site during periods when permitted activities are being conducted.

Sincerely,

Matthew R. Penski

Matthew R. Penski
Environmental Analyst I

Enclosures
cc: W. O'Brien, DOW
N.D. Eryou, PHD, PE
File

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

State Pollutant Discharge Elimination System (SPDES)
DISCHARGE PERMIT

First3.99

Industrial Code: 4581
Discharge Class (CL): 01
Toxic Class (TX): T
Major Drainage Basin: 17
Sub Drainage Basin: 01
Water Index Number:
Compact Area:

SPDES Number: NY0173703
DEC Number: 1-4728-00783/00003
Effective Date (EDP): 9/01/2008
Expiration Date (ExDP): 9/1/2013
Modification Dates:(EDPM) 11/1/2009

This SPDES permit is issued in compliance with Title 8 of Article 17 of the Environmental Conservation Law of New York State and in compliance with the Clean Water Act, as amended, (33 U.S.C. §1251 et.seq.)(hereinafter referred to as "the Act").

PERMITTEE NAME AND ADDRESS

Name: Long Island Macarthur Airport
Street: 100 Arrival Avenue
City: Ronkonkoma

Attention: Robert A. Schneider

State: NY Zip Code: 11779
is authorized to discharge from the facility described below:

FACILITY NAME AND ADDRESS

Name: Long Island Macarthur Airport
Location (C,T,V): Islip (T) County: Suffolk
Facility Address: 100 Arrival Avenue
City: Ronkonkoma State: NY Zip Code: 11779
NYTM -E: 660.2 NYTM - N: 4517.7
From Outfall No.: 001-003 at Latitude: 40 ° 47 ″ 23 ′ & Longitude: 73 ° 06 ″ 71 ′
into receiving waters known as: Groundwater Class: GA
and; (list other Outfalls, Receiving Waters & Water Classifications)

in accordance with: effluent limitations; monitoring and reporting requirements; other provisions and conditions set forth this permit; and 6 NYCRR Part 750-1.2(a) and 750-2.

DISCHARGE MONITORING REPORT (DMR) MAILING ADDRESS

Mailing Name: Long Island Macarthur Airport
Street: 100 Arrival Avenue
City: Ronkonkoma State: NY Zip Code: 11779
Responsible Official or Agent: Robert A. Schneider Phone: 631-467-3300

This permit and the authorization to discharge shall expire on midnight of the expiration date shown above and the permittee shall not discharge after the expiration date unless this permit has been renewed, or extended pursuant to law. To be authorized to discharge beyond the expiration date, the permittee shall apply for permit renewal not less than 180 days prior to the expiration date shown above.

DISTRIBUTION:

CO BWP - Permit Coordinator
RWE
RPA
J.Meyers, SCDHS

Permit Administrator: John A. Wieland, Deputy Permit Administrator
Address:NYSDEC
SUNY@Stony Brook
50 Circle Road
Stony Brook, New York, 11790-3409
Signature: Date: 09 / 22 / 2009

PERMIT LIMITS, LEVELS AND MONITORING DEFINITIONS

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OUTFALL	WASTEWATER TYPE	RECEIVING WATER	EFFECTIVE	EXPIRING
	This cell describes the type of wastewater authorized for discharge. Examples include process or sanitary wastewater, storm water, non-contact cooling water.	This cell lists classified waters of the state to which the listed outfall discharges.	The date this page starts in effect. (e.g. EDP or EDPM)	The date this page is no longer in effect. (e.g. ExDP)

PARAMETER	MINIMUM	MAXIMUM	UNITS	SAMPLE FREQ.	SAMPLE TYPE
e.g. pH, TRC, Temperature, D.O.	The minimum level that must be maintained at all instants in time.	The maximum level that may not be exceeded at any instant in time.	SU, °F, mg/l, etc.		

PARA-METER	EFFLUENT LIMIT	PRACTICAL QUANTITATION LIMIT (PQL)	ACTION LEVEL	UNITS	SAMPLE FREQUENCY	SAMPLE TYPE
	Limit types are defined below in Note 1. The effluent limit is developed based on the more stringent of technology-based standards, required under the Clean Water Act, or New York State water quality standards. The limit has been derived based on existing assumptions and rules. These assumptions include receiving water hardness, pH and temperature; rates of this and other discharges to the receiving stream; etc. If assumptions or rules change the limit may, after due process and modification of this permit, change.	For the purposes of compliance assessment, the analytical method specified in the permit shall be used to monitor the amount of the pollutant in the outfall to this level, provided that the laboratory analyst has complied with the specified quality assurance/quality control procedures in the relevant method. Monitoring results that are lower than this level must be reported, but shall not be used to determine compliance with the calculated limit. This PQL can be neither lowered nor raised without a modification of this permit.	Type I or Type II Action Levels are monitoring requirements, as defined below in Note 2, that trigger additional monitoring and permit review when exceeded.	This can include units of flow, pH, mass, Temperature, concentration. Examples include µg/l, lbs/d, etc.	Examples include Daily, 3/week, weekly, 2/month, monthly, quarterly, 2/yr and yearly.	Examples include grab, 24 hour composite and 3 grab samples collected over a 6 hour period.

Note 1: DAILY DISCHARGE: The discharge of a pollutant measured during a calendar day or any 24-hour period that reasonably represents the calendar day for the purposes of sampling. For pollutants expressed in units of mass, the 'daily discharge' is calculated as the total mass of the pollutant discharged over the day. For pollutants with limitations expressed in other units of measurement, the 'daily discharge' is calculated as the average measurement of the pollutant over the day. DAILY MAX: The highest allowable daily discharge. DAILY MIN: The lowest allowable daily discharge. MONTHLY AVG (daily avg): The highest allowable average of daily discharges over a calendar month, calculated as the sum of each of the daily discharges measured during a calendar month divided by the number of daily discharges measured during that month. RANGE: The minimum and maximum instantaneous measurements for the reporting period must remain between the two values shown. 7 DAY ARITHMETIC MEAN (7 day average): The highest allowable average of daily discharges over a calendar week. 12 MRA (twelve month rolling avg): The average of the most recent twelve month's monthly averages. 30 DAY GEOMETRIC MEAN (30 d geo mean): The highest allowable geometric mean of daily discharges over a calendar month, calculated as the antilog of : the sum of the log of each of the daily discharges measured during a calendar month divided by the number of daily discharges measured during that month. 7 DAY GEOMETRIC MEAN (7 d geo mean): The highest allowable geometric mean of daily discharges over a calendar week.

Note 2: ACTION LEVELS: Routine Action Level monitoring results, if not provided for on the Discharge Monitoring Report (DMR) form, shall be appended to the DMR for the period during which the sampling was conducted. If the additional monitoring requirement is triggered as noted below, the permittee shall undertake a short-term, high-intensity monitoring program for the parameter(s). Samples identical to those required for routine monitoring purposes shall be taken on each of at least three consecutive operating and discharging days and analyzed. Results shall be expressed in terms of both concentration and mass, and shall be submitted no later than the end of the third month following the month when the additional monitoring requirement was triggered. Results may be appended to the DMR or transmitted under separate cover to the same address. If levels higher than the Action Levels are confirmed, the permit may be reopened by the Department for consideration of revised Action Levels or effluent limits. The permittee is not authorized to discharge any of the listed parameters at levels which may cause or contribute to a violation of water quality standards. TYPE I : The additional monitoring requirement is triggered upon receipt by the permittee of any monitoring results in excess of the stated Action Level. TYPE II: The additional monitoring requirement is triggered upon receipt by the permittee of any monitoring results that show the stated action level exceeded for four of six consecutive samples, or for two of six consecutive samples by 20 % or more, or for any one sample by 50 % or more.

PERMIT LIMITS, LEVELS AND MONITORING

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OUTFALL No.	WASTEWATER TYPE	RECEIVING WATER	EFFECTIVE	EXPIRING
001	Stormwater from Tank Farm Area	Groundwater	11/1/2009	9/1/2013

PARAMETER	MINIMUM	MAXIMUM	UNITS	SAMPLE FREQUENCY	SAMPLE TYPE	FOOTNOTES (FN)
pH	5.5	8.8	SU	Monthly	Grab	

PARAMETER	COMPLIANCE LIMIT		MONITORING ACTION LEVEL		UNITS	SAMPLE FREQUENCY	SAMPLE TYPE	FN
				TYPE II				
Oil & Grease	NA	15			mg/l	Batch	Grab	
Benzene	NA	0.7			ug/l	Batch	Grab	
Toluene	NA	5			ug/l	Batch	Grab	
Xylene	NA	5			ug/l	Batch	Grab	1
Ethylbenzene	NA	5			ug/l	Batch	Grab	

NOTES:
1. Applicable to each isomer: m, o and p individually.

OUTFALL No.	WASTEWATER TYPE	RECEIVING WATER	EFFECTIVE	EXPIRING
002	Stormwater from Ramp Areas	Groundwater	11/1/2009	9/1/2013

PARAMETER	MINIMUM	MAXIMUM	UNITS	SAMPLE FREQUENCY	SAMPLE TYPE	FOOTNOTES (FN)
pH	5.5	8.8	SU	Monthly	Grab	

PARAMETER	COMPLIANCE LIMIT		MONITORING ACTION LEVEL		UNITS	SAMPLE FREQUENCY	SAMPLE TYPE	FN
				TYPE II				
Oil & Grease	NA	15			mg/l	Monthly	Grab	
Benzene	NA	0.7			ug/l	Monthly	Grab	
Toluene	NA	5			ug/l	Monthly	Grab	
Xylene	NA	5			ug/l	Monthly	Grab	1
Ethylbenzene	NA	5			ug/l	Monthly	Grab	
Propylene Glycol	NA	1000			ug/l	Monthly	Grab	2

NOTES:

1. Applicable to each isomer: m, o and p individually.
2. Propylene Glycol samples are to be collected from downgradient monitoring wells MW1 & MW2. These samples are to be collected monthly from November to April. Prior to the first deicing season the permittee shall purge and sample the groundwater monitoring wells on three consecutive days for Propylene Glycol. These sample results are to be submitted to this Department to determine the ambient levels

OUTFALL No.	WASTEWATER TYPE	RECEIVING WATER	EFFECTIVE	EXPIRING
003	Sanitary Discharges	Groundwater	9/1/2008	9/1/2013

NO MONITORING REQUIRED

SPECIAL CONDITIONS - INDUSTRY BEST MANAGEMENT PRACTICES

1. **General** - The permittee shall develop, maintain, and implement a Best Management Practices (BMP) plan to prevent releases of significant amounts of pollutants to the waters of the State through plant site runoff; spillage and leaks; sludge or waste disposal; and stormwater discharges including, but not limited to, drainage from raw material storage.

   The BMP plan shall be documented in narrative form and shall include the 13 minimum BMPs and any necessary plot plans, drawings, or maps. Other documents already prepared for the facility such as a Safety Manual or a Spill Prevention, Control and Countermeasure (SPCC) plan may be used as part of the plan and may be incorporated by reference. A copy of the current BMP plan shall be submitted to the Department as required in item (2.) below and a copy must be maintained at the facility and shall be available to authorized Department representatives upon request.

2. **Compliance Deadlines** - The initial completed BMP plan shall be submitted within six months of effective date of this permit to the Regional Water Engineer. The BMP plan shall be implemented within 6 months of submission, unless a different time frame is approved by the Department. The BMP plan shall be reviewed annually and shall be modified whenever: (a) changes at the facility materially increase the potential for releases of pollutants; (b) actual releases indicate the plan is inadequate, or (c) a letter from the Department identifies inadequacies in the plan. The permittee shall certify in writing, as an attachment to the December Discharge Monitoring Report (DMR), that the annual review has been completed. All BMP plan revisions (with the exception of SWPPPs - see item (4.B.) below) must be submitted to the Regional Water Engineer within 30 days. Note that the permittee is not required to obtain Department approval of the BMP plan (or of any SWPPPs) unless notified otherwise. Subsequent modifications to or renewal of this permit does not reset or revise these deadlines unless a new deadline is set explicitly by such permit modification or renewal.

3. **Facility Review** - The permittee shall review all facility components or systems (including but not limited to material storage areas; in-plant transfer, process, and material handling areas; loading and unloading operations; storm water, erosion, and sediment control measures; process emergency control systems; and sludge and waste disposal areas) where materials or pollutants are used, manufactured, stored or handled to evaluate the potential for the release of pollutants to the waters of the State. In performing such an evaluation, the permittee shall consider such factors as the probability of equipment failure or improper operation, cross-contamination of storm water by process materials, settlement of facility air emissions, the effects of natural phenomena such as freezing temperatures and precipitation, fires, and the facility's history of spills and leaks. The relative toxicity of the pollutant shall be considered in determining the significance of potential releases.

   The review shall address all substances present at the facility that are identified in Tables 6-10 of SPDES application Form NY-2C (available at http://www.dec.ny.gov/docs/permits_ej_operations_pdf/form2c.pdf) or that are required to be monitored for by the SPDES permit.

4. A. **13 Minimum BMPs** - Whenever the potential for a release of pollutants to State waters is determined to be present, the permittee shall identify BMPs that have been established to prevent or minimize such potential releases. Where BMPs are inadequate or absent, appropriate BMPs shall be established. In selecting appropriate BMPs, the permittee shall consider good industry practices and, where appropriate, structural measures such as secondary containment and erosion/sediment control devices and practices. USEPA guidance for development of stormwater elements of the BMP is available in the September 1992 manual *Storm Water Management for Industrial Activities,* EPA 832-R-92-006 (available from NTIS, 703-487-4650, order # PB 92235969). As a minimum, the plan shall include the following BMPs:

   1. BMP Pollution Prevention Team
   2. Reporting of BMP Incidents
   3. Risk Identification & Assessment
   4. Employee Training
   5. Inspections and Records
   6. Security
   7. Preventive Maintenance
   8. Good Housekeeping
   9. Materials/Waste Handling, Storage, & Compatibility
   10. Spill Prevention & Response
   11. Erosion & Sediment Control
   12. Management of Runoff
   13. Street Sweeping

   Note that for some facilities, especially those with few employees, some of the above BMPs may not be applicable. It is acceptable in these cases to indicate "Not Applicable" for the portion(s) of the BMP Plan that do not apply to your facility, along with an explanation.

B. **Stormwater Pollution Prevention Plans (SWPPPs) Required for Discharges of Stormwater From Construction Activity to Surface Waters** - As part of BMP #11, a SWPPP shall be developed prior to the initiation of any site disturbance of one acre or more of uncontaminated area. Uncontaminated area means soils or groundwater which are free of contamination by any toxic or non-conventional pollutants identified in Tables 6-10 of SPDES application Form NY-2C. Disturbance of any size contaminated area(s) and the resulting discharge of contaminated stormwater is not authorized by this permit unless the discharge is under State or Federal oversight as part of a remedial program or after review by the Regional Water Engineer; nor is such discharge authorized by any SPDES general permit for stormwater discharges. SWPPPs are not required for discharges of stormwater from construction activity to groundwaters.

The SWPPP shall conform to the *New York Standards and Specifications for Erosion and Sediment Control* and *New York State Stormwater Management Design Manual,* unless a variance has been obtained from the Regional Water Engineer, and to any local requirements. The permittee shall submit a copy of the SWPPP and any amendments thereto to the local governing body and any other authorized agency having jurisdiction or regulatory control over the construction activity **at least 30 days prior to soil disturbance.** The SWPPP shall also be submitted to the Regional Water Engineer if contamination, as defined above, is involved and the permittee must obtain a determination of any SPDES permit modifications and/or additional treatment which may be required prior to soil disturbance. Otherwise, the SWPPP shall be submitted to the Department only upon request. When a SWPPP is required, a properly completed *Notice of Intent* (NOI) form shall be submitted (available at www.dec.ny.gov/chemical/43133.html) prior to soil disturbance. Note that submission of a NOI is required for informational purposes; the permittee is not eligible for and will not obtain coverage under any SPDES general permit for stormwater discharges, nor are any additional permit fees incurred. SWPPPs must be developed and submitted for subsequent site disturbances in accordance with the above requirements. The permittee is responsible for ensuring that the provisions of each SWPPP are properly implemented.

5. **Facilities with Petroleum and/or Chemical Bulk Storage (PBS and CBS) Areas** - Compliance must be maintained with all applicable regulations including those involving releases, registration, handling and storage (6NYCRR 595-599 and 612-614). Stormwater discharges from handling and storage areas should be eliminated where practical.

A. Spill Cleanup - All spilled or leaked substances must be removed from secondary containment systems as soon as practical and for CBS storage areas within 24 hours, unless written authorization is received from the Department . The containment system must be thoroughly cleaned to remove any residual contamination which could cause contamination of stormwater and the resulting discharge of pollutants to waters of the State. Following spill cleanup the affected area must be completely flushed with clean water three times and the water removed after each flushing for proper disposal in an on-site or off-site wastewater treatment plant designed to treat such water and permitted to discharge such wastewater. Alternately, the permittee may test the first batch of stormwater following the spill cleanup to determine discharge acceptability. If the water contains no pollutants it may be discharged. Otherwise it must be disposed of as noted above. See *Discharge Monitoring* below for the list of parameters to be sampled for.

B. Discharge Operation - Stormwater must be removed before it compromises the required containment system capacity. Each discharge may only proceed with the prior approval of the permittee staff person responsible for ensuring SPDES permit compliance. Bulk storage secondary containment drainage systems must be locked in a closed position except when the operator is in the process of draining accumulated stormwater. Transfer area secondary containment drainage systems must be locked in a closed position during all transfers and must not be reopened unless the transfer area is clean of contaminants. Stormwater discharges from secondary containment systems should be avoided during periods of precipitation. A logbook shall be maintained on site noting the date, time and personnel supervising each discharge.

C. Discharge Screening - Prior to each discharge from a secondary containment system the stormwater must be screened for contamination*. All stormwater must be inspected for visible evidence of contamination. Additional screening methods shall be developed by the permittee as part of the overall BMP Plan, e.g. the use of volatile gas meters to detect the presence of gross levels of gasoline or volatile organic compounds. If the screening indicates contamination, the permittee must collect and analyze a representative sample** of the stormwater. If the water contains no pollutants it may be discharged. Otherwise it must either be disposed of in an on site or off site wastewater treatment plant designed to treat and permitted to discharge such wastewater or the Regional Water Engineer can be contacted to determine if it may be discharged without treatment.

D. Discharge Monitoring - Unless the discharge from any bulk storage containment system outlet is identified in the SPDES permit as an outfall with explicit effluent and monitoring requirements, the permittee shall monitor the outlet as follows:

(i) *Bulk Storage Secondary Containment Systems:*

(a) The volume of each discharge from each outlet must be monitored. Discharge volume may be calculated by measuring the depth of water within the containment area times the wetted area converted to gallons or by other suitable methods. A representative sample shall be collected of the first discharge* following any cleaned up spill or leak. The sample must be analyzed for pH, the substance(s) stored within the containment area and any other pollutants the permittee knows or has reason to believe are present**.

(b) Every fourth discharge* from each outlet must be sampled for pH, the substance(s) stored within the containment area and any other pollutants the permittee knows or has reason to believe are present**.

(ii) *Transfer Area Secondary Containment Systems:*

The first discharge* following any spill or leak must be sampled for flow, pH, the substance(s) transferred in that area and any other pollutants the permittee knows or has reason to believe are present**.

6. **Airports:** The BMP plan must include pertinent elements of the SWPPP for industrial activities.
The requirements listed under this section apply to stormwater discharges associated with industrial activity from air transportation facilities including air transportation (scheduled and non-scheduled); air courier services; airports; flying fields (except those maintained by aviation clubs); air terminal services including air traffic control (except government); aircraft storage at airports; aircraft upholstery repair; airfreight handling at airports; airport hangar rental; airport leasing, if operating airport; airport terminal services; hangar operation; airport, aircraft service and maintenance including aircraft cleaning and janitorial service; aircraft servicing /repairing except on a factory basis); vehicle maintenance shops; material handling facilities; equipment clearing operations; and airport/aircraft deicing and anti-icing. [Note: For the purpose of this section, the term "deicing" is defined as the process to remove frost, snow, or ice and "anti-icing" is the process which prevents the accumulation of frost, snow, or ice.] Only those portions of the facility that are either involved in vehicle maintenance (including vehicle rehabilitation, mechanical repairs, painting, fueling, and lubrication), equipment cleaning operations, or deicing/anti-icing operations are addressed under this section. Tenants and/or other entities that apply or otherwise use deicing and/or anti-icing materials shall provide all necessary information to the permittee for the permittee to complete all requirements under this section.

Additional Requirements for the BMP Plan: BMPs shall be developed for areas of the facility occupied by tenants of the airport and shall be integrated with the BMP plan for the entire airport. For the purposes of this permit, tenants of the airport facility include airline passenger or cargo companies, fixed based operators and other parties who have contracts with the airport authority to conduct business operations on airport property and whose operations result in stormwater discharges associated with industrial activity. The BMP plan shall include, at a minimum, the following items.

A. Site description

(i) Site map - The site map shall identify where any of the following activities may be exposed to precipitation/surface runoff: aircraft and runway deicing/anti-icing operations; fueling stations; aircraft, ground vehicle and equipment maintenance/cleaning areas; and storage areas for aircraft, ground vehicles and equipment awaiting maintenance.

(ii) Summary of potential pollutant sources - A narrative description of the potential pollutant sources from the following activities: aircraft, runway, ground vehicle and equipment maintenance and cleaning; aircraft and runway deicing/anti-icing operations (including apron and centralized aircraft deicing/anti-icing stations, runways, taxiways and ramps). Facilities which conduct deicing/anti-icing operations shall maintain a record of the types (including the Material Safety Data Sheets (MSDS)) and monthly quantities of deicing/anti-icing chemicals used, either as measured amounts, or in the absence of metering, as estimated amounts. This includes all deicing/anti-icing chemicals, not just glycols and urea (e.g., potassium acetate). Tenants and fixed-base operators who conduct deicing/anti-icing operations shall provide the above information to the airport authority for inclusion in the BMP for the entire facility.

B. Stormwater controls

(i) Good housekeeping

(a) Aircraft, ground vehicle and equipment maintenance areas - The permittee must describe and implement measures that prevent or minimize the contamination of stormwater runoff from all areas used for aircraft, ground vehicle and equipment maintenance (including the maintenance conducted on the terminal apron and in dedicated hangars). The following practices (or their equivalents) shall be considered: performing maintenance activities indoors; maintaining an organized inventory of materials used in the maintenance areas ; draining all parts of fluids prior to disposal; preventing the practice of hosing down the apron or hangar floor; using dry cleanup methods ; and collecting the stormwater runoff from the maintenance area and providing treatment or recycling.

the drains, etc.) to prevent these materials from later becoming a source of stormwater contamination. Used deicing fluid should be recycled whenever possible.

(iv) Routine facility inspections - The inspection frequency shall be specified in the plan. At a minimum, inspections shall be conducted once per month during deicing/anti-icing season (e.g., October through April for most airports). If deicing occurs before or after this period, the inspections shall be expanded to include all months during which deicing chemicals may be used. Also, if significantly or deleteriously large quantities of deicing chemicals are being spilled or discharged, or if water quality impacts have been reported, the inspection frequency shall be increased to weekly until such time as the chemical spills/discharges or impacts are reduced to acceptable levels.

(v) Comprehensive site compliance evaluation - The annual site compliance evaluations shall be conducted by qualified facility personnel during periods of actual deicing operations, if possible. If not practicable during active deicing or if the weather is too inclement, the evaluations shall be conducted when deicing operations are likely to occur and the materials and equipment for deicing are in place.

E. Discharge Reporting - Any results of monitoring required above, excluding screening data, must be submitted to the Department by appending them to the corresponding DMR. Failure to perform the required discharge monitoring and reporting shall constitute a violation of the terms of the SPDES permit.

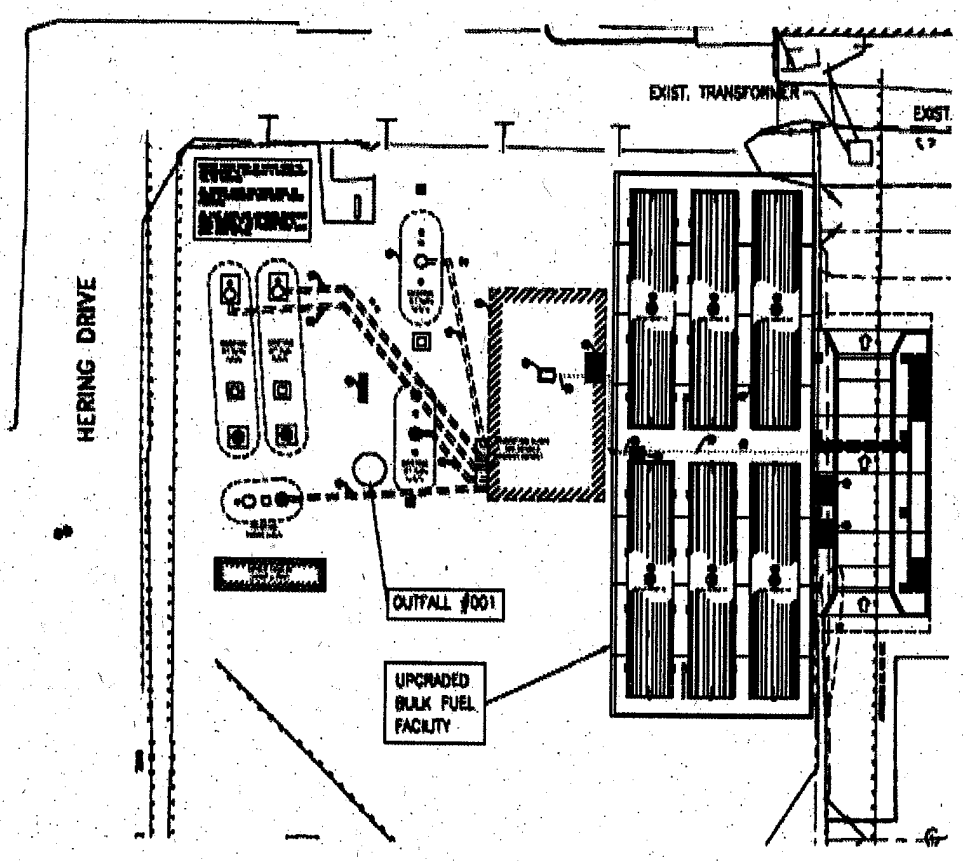
F. Prohibited Discharges - **In all cases, any discharge which contains a visible sheen, foam, or odor, or may cause or contribute to a violation of water quality is prohibited.** The following discharges are prohibited unless specifically authorized elsewhere in this SPDES permit: spills or leaks, tank bottoms, maintenance wastewaters, wash waters where detergents or other chemicals have been used, tank hydrotest and ballast waters, contained fire fighting runoff, fire training water contaminated by contact with pollutants or containing foam or fire retardant additives, and unnecessary discharges of water or wastewater into secondary containment systems.

* Discharge includes stormwater discharges and snow and ice removal. If applicable, a representative sample of snow and/or ice should be collected and allowed to melt prior to assessment.

** If the stored substance is gasoline or aviation fuel then sample for oil & grease, benzene, ethylbenzene, naphthalene, toluene and total xylenes (EPA method 602). If the stored substance is kerosene, diesel fuel, fuel oil, or lubricating oil then sample for oil & grease and polynuclear aromatic hydrocarbons (EPA method 610). If the substance(s) are listed in Tables 6-8 of SPDES application form NY-2C then sampling is required. If the substance(s) are listed in NY-2C Tables 9-10 sampling for appropriate indicator parameters may be required, e.g. BOD5 or toxicity testing. Contact the facility inspector for further guidance. In all cases flow and pH monitoring is required.

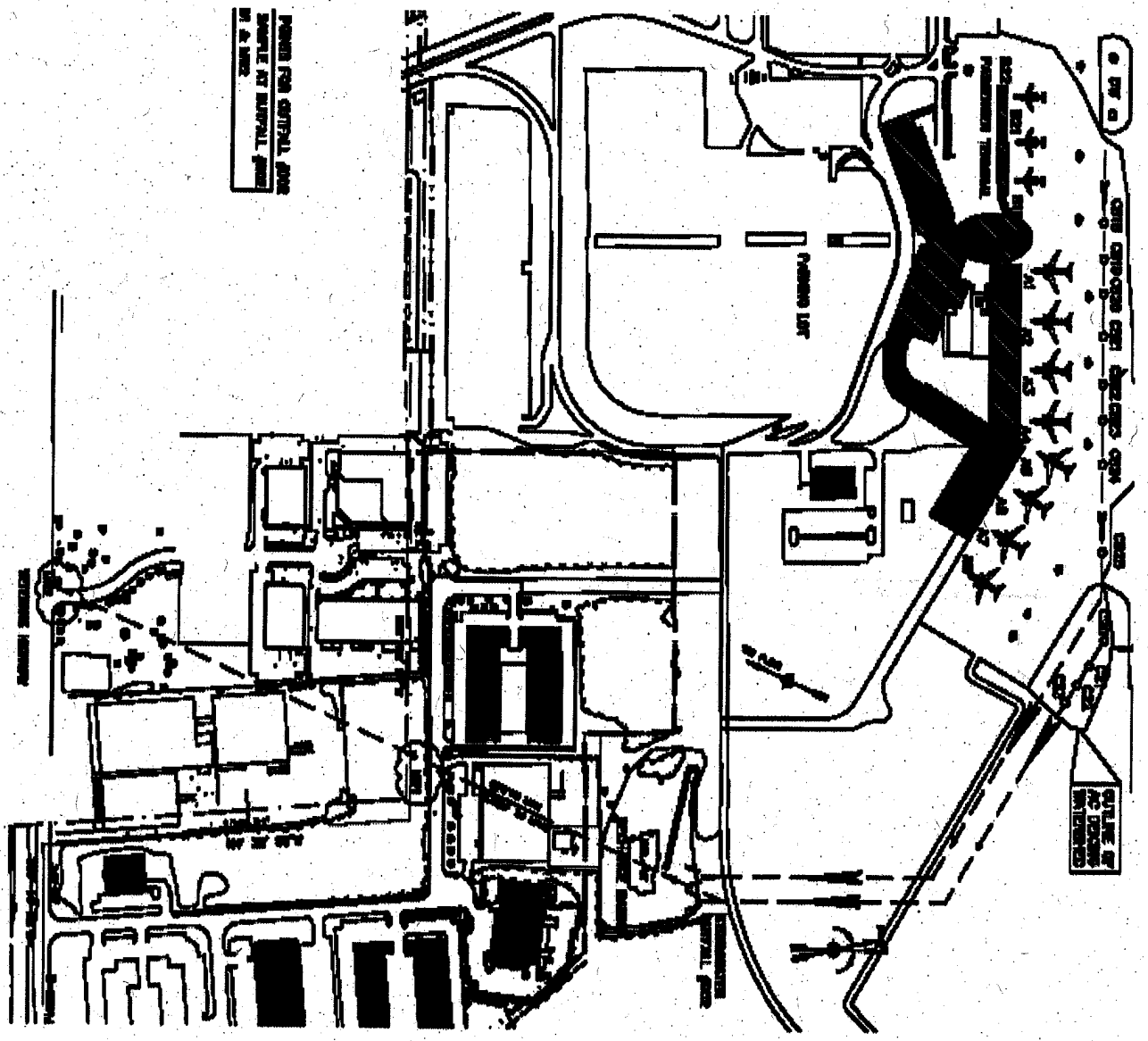
MONITORING LOCATIONS

The permittee shall take samples and measurements, to comply with the monitoring requirements specified in this permit, at the location(s) specified below: Outfall #001



MONITORING LOCATIONS

The permittee shall take samples and measurements, to comply with the monitoring requirements specified in this permit, at the location(s) specified below: Outfall # 002



RECORDING, REPORTING AND ADDITIONAL MONITORING REQUIREMENTS

a) The permittee shall also refer to 6 NYCRR Part 750-1.2(a) and 750-2 for additional information concerning monitoring and reporting requirements and conditions.

b) The monitoring information required by this permit shall be summarized, signed and retained for a period of five years from the date of the sampling for subsequent inspection by the Department or its designated agent. **Also, monitoring information required by this permit shall be summarized and reported by submitting;**

[X] (if box is checked) completed and signed Discharge Monitoring Report (DMR) forms for each 1 month reporting period to the locations specified below. Blank forms are available at the Department's Albany office listed below. The first reporting period begins on the effective date of this permit and the reports will be due no later than the 28th day of the month following the end of each reporting period.

[ ] (if box is checked) an annual report to the Regional Water Engineer at the address specified below. The annual report is due by February 1 and must summarize information for January to December of the previous year in a format acceptable to the Department.

[ ] (if box is checked) a monthly "Wastewater Facility Operation Report..." (form 92-15-7) to the:

[ ] Regional Water Engineer and/or [ ] County Health Department or Environmental Control Agency specified below

Send the DMRs with **original signatures** to:

Department of Environmental Conservation
Division of Water
Bureau of Water Compliance Programs
625 Broadway
Albany, New York 12233-3506

Phone: (518) 402-8177

Send a **copy** of each DMR page to:

Department of Environmental Conservation
Regional Water Engineer
SUNY @ Stony Brook
50 Circle Road
Stony Brook, New York, 11790-3409

Phone: (631) 444-0405

c) Noncompliance with the provisions of this permit shall be reported to the Department as prescribed in 6 NYCRR Part 750-1.2(a) and 750-2.

d) Monitoring must be conducted according to test procedures approved under 40 CFR Part 136, unless other test procedures have been specified in this permit.

e) If the permittee monitors any pollutant more frequently than required by the permit, using test procedures approved under 40 CFR Part 136 or as specified in this permit, the results of this monitoring shall be included in the calculations and recording of the data on the Discharge Monitoring Reports.

f) Calculation for all limitations which require averaging of measurements shall utilize an arithmetic mean unless otherwise specified in this permit.

g) Unless otherwise specified, all information recorded on the Discharge Monitoring Report shall be based upon measurements and sampling carried out during the most recently completed reporting period.

h) Any laboratory test or sample analysis required by this permit for which the State Commissioner of Health issues certificates of approval pursuant to section five hundred two of the Public Health Law shall be conducted by a laboratory which has been issued a certificate of approval. Inquiries regarding laboratory certification should be sent to the Environmental Laboratory Accreditation Program, New York State Health Department Center for Laboratories and Research, Division of Environmental Sciences , The Nelson A. Rockefeller Empire State Plaza, Albany, New York 12201.

Appendix B

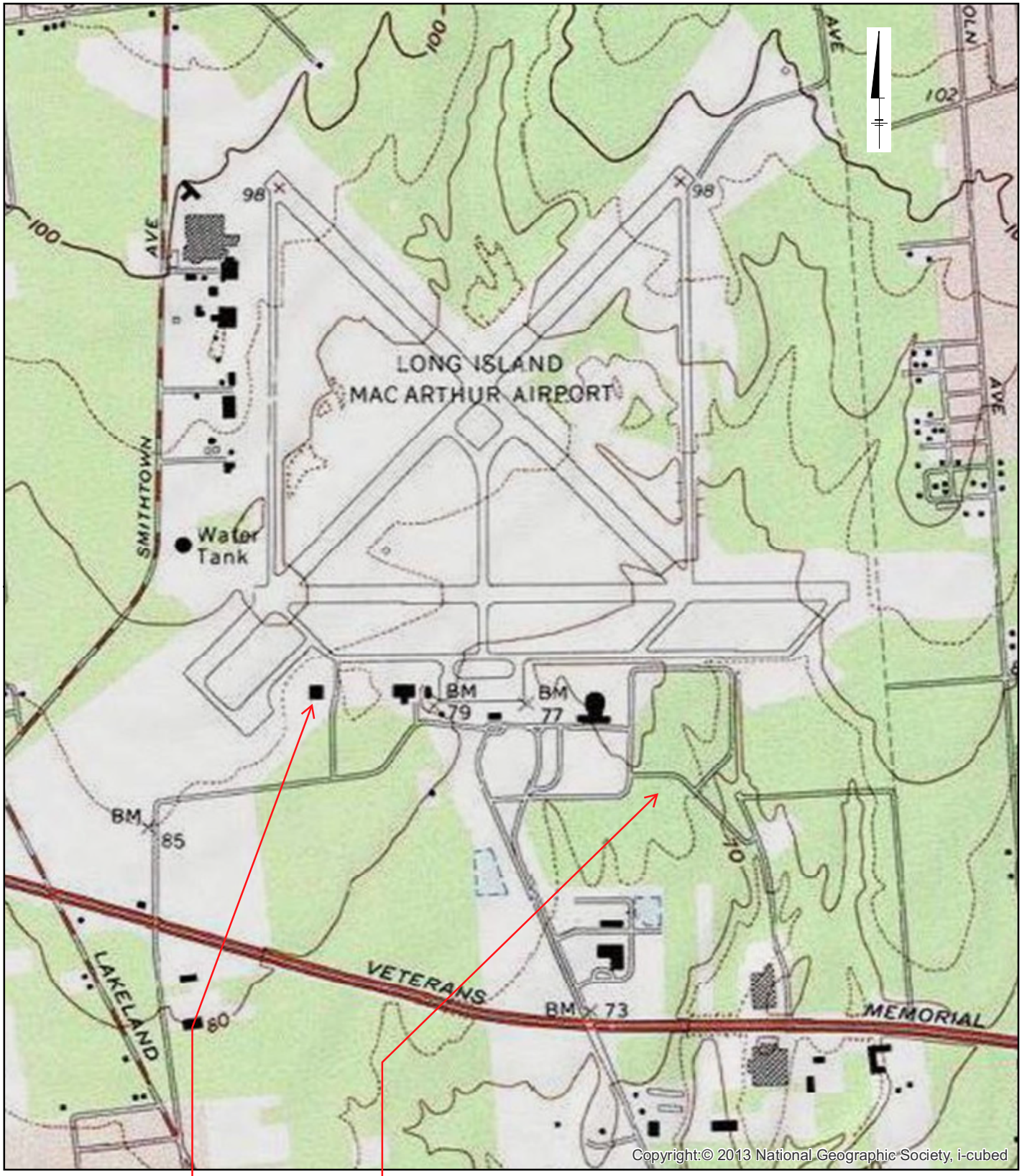
Location and Site Maps

Figure 1. Airport Location Map

Figure 2. Existing Airport Layout Plan

Figure 3. Drainage Areas

Figure 4. Glycol Pre-treatment System



OUTFALL #001

OUTFALL #002

0 0.050.1 0.2 0.3 0.4 Miles

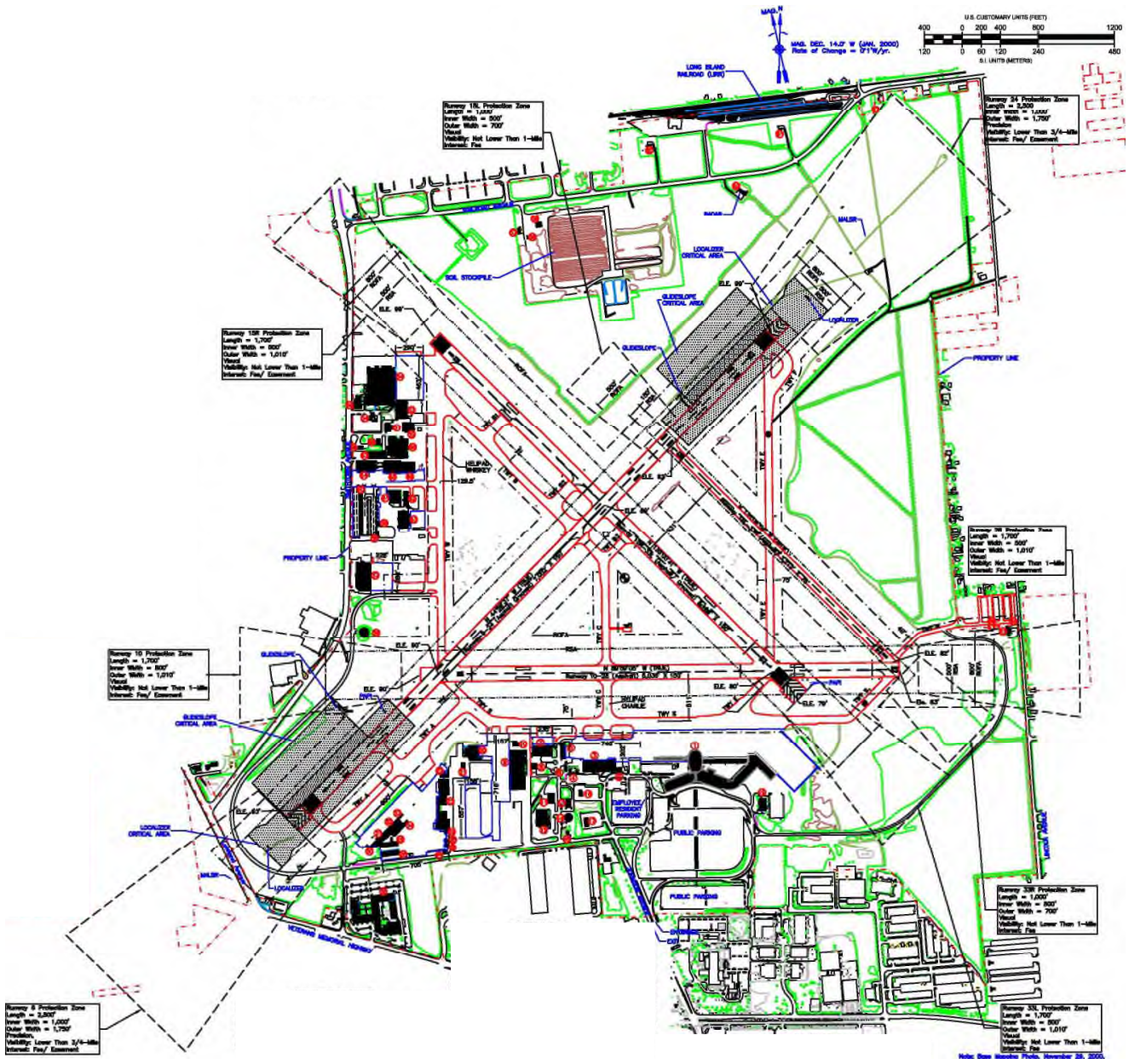
MACARTHUR AIRPORT
RONKONKOMA, NY

AIRPORT LOCATOR MAP

ARCADIS

FIGURE
1

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BUILDING LEGEND

BLDG. NO.	DESCRIPTION
1	Terminal
2	L1 Jet Center
3	L1 Jet Center
4	FAA Air Traffic Control Tower (ATCT)
5	Airport Rescue and Firefighting (ARFF)
6	Lighting Vault
7	L1 Jet Center (SFI North)
8	L1 Jet Center Maintenance
9	New York Army National Guards (NYANG)
10	New York Army National Guards (NYANG)
11	Maintenance Storage Bld.
12	Airport Maintenance Bld.
13	Air Freight Bld.
14	Hotel Storage
15	Maintenance/Fuel Bld.
16	Rental Car Facilities
17	Rental Car Facilities
18	Rental Car Facilities
19	FAA APOC
20	Fixed Air
21	Jet Center Fuel Farm
22	Mid Island Fuel Farm
23	Mid Island
24	Mid Island T-Hangar (1-unit)
25	Mid Island T-Hangar (1-unit)
26	Mid Island T-Hangar (1-unit)
27	Mid Island T-Hangar (1-unit)
28	Mid Island Hangar
29	Mid Island Hangar
30	Mid Island T-Hangar (1-unit)
31	Mid Island Hangar
32	Mid Island Hangar
33	Mid Island Hangar
34	Mid Island Hangar
35	Hotel (Holiday Inn)
36	Pump House and Reservoir
37	L1 Jet Center (ATC)
38	Private Hangar
39	Private Hangar
40	L1 Jet Center T-Hangars (10 units)
41	L1 Jet Center
42	Insulated Tote
43	Hangar 1 (U.S. Customs)
44	ANP Maintenance/Rescue Helicopters
45	L1 Jet Center (SFI North)
46	L1 Jet Center (SFI North)
47	L1 Jet Center
48	Vacant
49	Aero Trades
50	H&S Maintenance
51	Police
52	Police Hangar
53	Aero Trades
54	Gemalt
55	Gemalt

BUILDING LEGEND CONT

BLDG. NO.	DESCRIPTION
56	Abandoned Basement
57	Compost Facility
58	Compost Facility
59	Compost Facility
60	FAA Transmitter Site
61	ASDO
62	FAA ASR-9 Radar
63	Old Air Patrol (OAP) Bld.
64	Private T-Hangars (10 units)
65	Private T-Hangars (10 units)

SYMBOL LEGEND

EXISTING	DESCRIPTION
	PROPERTY LINE
	FENCE LINE
	CONTOUR LINE
10.00	SURFACE ELEVATION
	TREE LINE
	BUILDING ON AIRPORT
	BUILDING OFF AIRPORT
	RUNWAY OBJECT FREE AREA (ROFA)
	RUNWAY SAFETY AREA (RSA)
	RUNWAY PROTECTION ZONE (RPZ)
	AIRPORT REFERENCE POINT
	AIRPORT BEACON
	COMPASS CALIBRATION PAD
	PAPI
	APPROACH LIGHTING SYSTEM
	REIL
	STORMWATER DESIGNS SHOWN
	PRECISION APPROACH EQUIPMENT CRITICAL AREAS

LONG ISLAND MACARTHUR AIRPORT

EXISTING AIRPORT LAYOUT PLAN

ARCADIS

FIGURE 2

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SITE PLAN

DRAINAGE AREA	DELINEATION	DISCHARGE LOCATION
A		RECHARGE BASIN 1
B		RECHARGE BASIN 2
C		SWALE AREA 3
D		SWALE AREA 4
E		RECHARGE BASIN 5
F		SWALE AREA 6

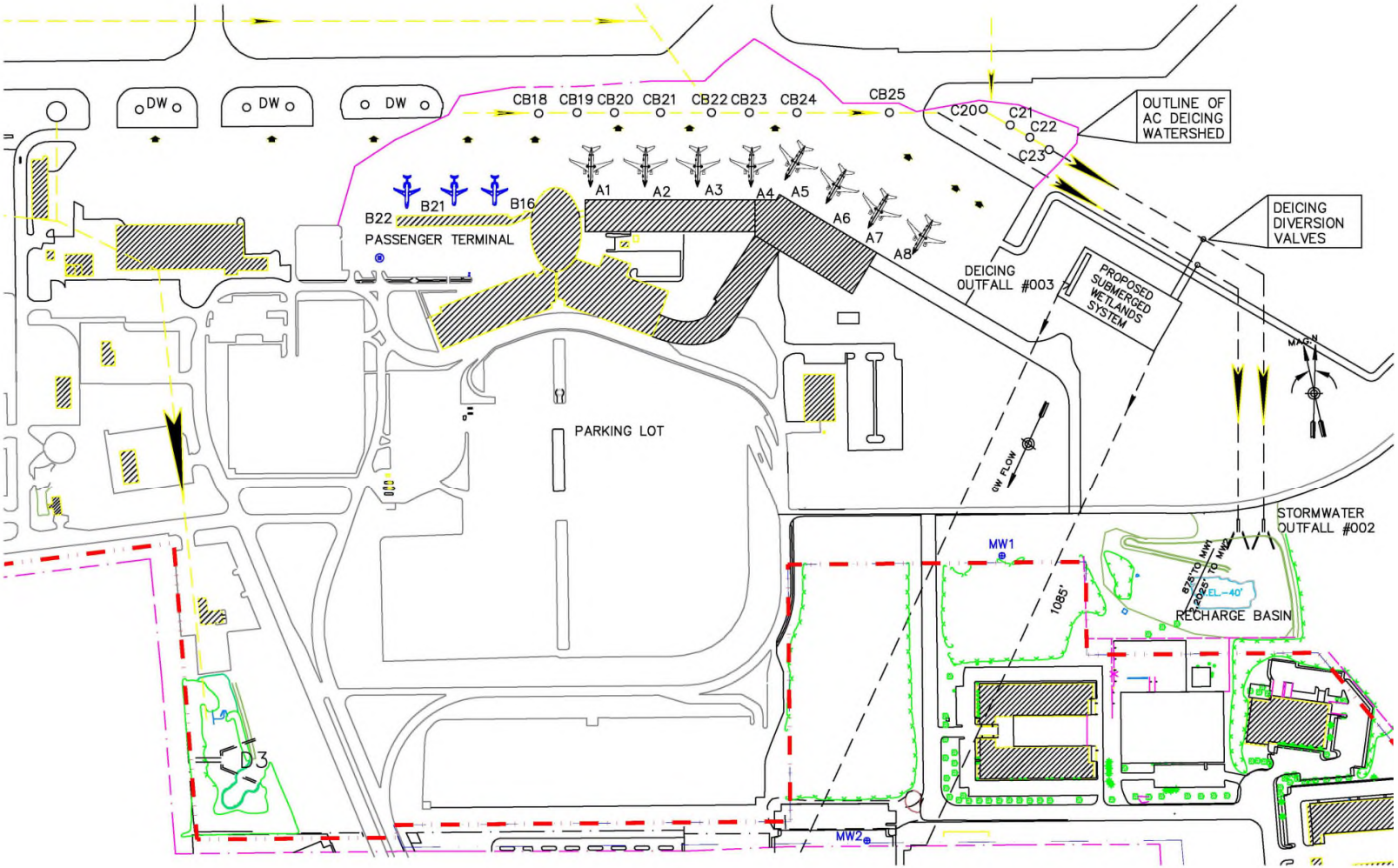
LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NY

DRAINAGE AREAS

ARCADIS

FIGURE
3

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AIRCRAFT DEICING AREA

NOT TO SCALE:

STORMWATER MONITORING POINTS FOR OUTFALL #002

HYDROCARBONS = GRAB SAMPLE AT OUTFALL #002
PROPYLENE GLYCOL = MW1 & MW2

LONG ISLAND MACARTHUR AIRPORT
RONKONKOMA, NY

GLYCOL PRETREATMENT SYSTEM

ARCADIS

FIGURE
4

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Appendix C

Potential Target Industrial Activities

LONG ISLAND MACARTHUR AIRPORT (ISP)

BEST MANAGEMENT PRACTICES – POTENTIAL TARGET INDUSTRIAL ACTIVITIES

Target Activities		RECOMMENDED STORMWATER BEST MANAGEMENT PRACTICE															
		BMP1	BMP2	BMP3	BMP4	BMP5	BMP6	BMP6	BMP7	BMP8	BMP9	BMP10	BMP11	BMP12	BMP13	BMP14	BMP15
AC Deicing	AD	X						X	X	X	X	X	X		X	X	
AC Fueling	AF		X					X	X	X	X	X	X		X		X
AC Maint	AM			X			X	X	X		X	X		X	X	X	
Grounds Maint	GM					X	X	X	X	X	X	X	X		X	X	
Chem Storage	CS						X	X	X	X	X	X	X	X	X		X
Equip Fueling	EF						X	X	X	X	X	X		X	X		
Equip. Maint	EM			X		X	X	X		X			X		X	X	
Fuel Storage	FS						X	X	X	X	X	X	X		X		
Vehicle Fuel	VF			X			X	X	X	X	X	X	X		X	X	
Vehicle Maint	VM			X				X	X	X		X	X		X	X	
Waste Mg't	WM						X	X	X	X	X	X	X		X	X	

X = Recommended BMPs for Target Industrial Activity

Kev to Best Management Practices (BMPs):

BMP I Aircraft Deicing
BMP 2 Aircraft, Vehicle & Equipment Fueling
BMP 3 Aircraft, Vehicle & Equipment Maintenance
BMP 4 Aircraft, Vehicle & Equipment Washing, Steam Cleaning & Degreasing
BMP 5 Building & Grounds Maintenance
BMP 6 Chemical & Fuel Storage and Handling
BMP 7 Elimination of Non-Stormwater Discharges to Maintenance Storm Drains
BMP 8 Spills Management
BMP 9 Oil/Water Separators
BMP 10 Outdoor Handling of Material
BMP 11 Outdoor Materials Storage
BMP 12 Waste Management
BMP 13 Stormwater Pollution Prevention Education
BMP 14 Sweeping & Maintenance
BMP 15 Security

Appendix D

Best Management Practices

Town of Islip
Long Island MacArthur Airport

BMP1 AIRCRAFT DEICING

PURPOSE:

Prevent or reduce the discharge of pollutants to soil, groundwater and/or stormwater from aircraft deicing and anti-icing procedures. The level of chemical pollutants associated with the discharge of deicing compounds to groundwater, can impact groundwater quality.

APPROACH TO EXISTING FACILITY ACTIVITIES:

Operational Considerations

- Personnel involved with deicing operations are trained. The training should, at a minimum, include source reduction techniques, best management practices, good housekeeping and should educate about the environmental impacts of over spraying.
- Depending on weather conditions, apply only enough fluid to surfaces to ensure the safe operation of the aircraft. Excess fluid dripped to the ground enters directly into the storm drainage system.
- Depending on weather conditions use a range of propylene glycol/water blends (i.e., warmer temperatures, use a 30/70 glycol/water blend, colder temperatures/inclement weather use a 55/45 mix) to minimize the discharge of contaminants.
- The purchase of new deicing trucks should include more efficient nozzles, heated deicing agent, blending capabilities and air.
- Consider using alternative low environmental-impact deicing/anti-icing compounds now under development, once approved by the FAA. Implement forthcoming recommendations of the FAA technical committee on deicing.
- Transfer deicing agents only in paved areas.
- The use of ethylene glycol deicing fluid is discouraged at ISP.

RELEVANT RULES AND REGULATIONS:

- 6 NYCRR Parts 595-599 Chemical Bulk Storage Regulations
- 6 NYCRR Parts 370-374 New York State Hazardous Waste Regulations
- 6 NYCRR Part 360 Solid Waste Regulations
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous Substance
- 40 CFR 122-124 NPDES Regulations for StormWater Discharges
- 40 CFR 302 Designation of Reportable Quantities and Notification Requirements for Hazardous Substances under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA)
- 40 CFR 372 Toxic Chemical Release Reporting: Community Right-to-Know
- 40 CFR 401 Effluent Limitation Guidelines

Town of Islip
Long Island MacArthur Airport

BMP2 AIRCRAFT, VEHICLE AND EQUIPMENT FUELING

PURPOSE:

Prevent or reduce the discharge of pollutants to stormwater, groundwater, soil, and air from aircraft, vehicle, and equipment fueling.

APPROACH TO EXISTING FACILITY ACTIVITIES:

Operational Considerations

General

- Install shear valves or "breakaway" hose connections at all fuel dispensing stations and on all fuel dispensing equipment that will provide emergency shutdown of flow should the fueling connection be broken through movement.
- Periodically check hoses associated with fuel dispensing for leaks and tears.
- Automatic shut-off mechanisms should be in place on fuel tankers. These valves should remain in the closed position unless manually opened during fueling.
- Use absorbent materials and spot cleaning for small spills; do not hose down the area unless the storm drains in close proximity are blocked and drainage is collected by vacuum vehicle and disposed of through a permitted connection to an approved treatment facility.
- Avoid mobile fueling of equipment wherever feasible; fuel mobile equipment at designated fueling areas. Use drain blockers (e.g., pigs/mats) at catch basins or install gate valves at catch basins for use during fueling activity.
- Collect and properly dispose of any fuel spilled or leaked. Vacuum equipment/vehicles are recommended for collection.
- Always dispose of materials in an approved manner. Never discharge materials to a catch basin.
- Employ secondary containment or cover when transferring fuel from a tank truck to a vehicle or equipment fuel tank. .
- Manage the disposal of water that collects in fuel tanks and fueling hydrant sumps according to state and federal regulations.
- Inspect, clean and maintain sumps and oil/water separators at appropriate intervals.

Motor Vehicle/Equipment

- Ensure that motor vehicle fuel composition meets seasonal requirements for oxygen content and volatility.
- Operate appropriate vapor recovery equipment at gasoline dispensing sites.
- Install berms or curbing to divert stormwater runoff away from fueling area to avoid contact with contaminated surfaces.

Operational Considerations (Continued)

- Provide appropriate monitoring for tanks containing fuel, such as: - Level indicators and gauges
  - Overfill protection with alarms
  - Interstitial leak detection for double-walled tanks
  - Routine inspection/lockout for drainage valves for tank containment areas.
- Test spill prevention/overfill protection equipment annually.
- Fuel pumps intended for vehicular use should be posted with signs stating 'No Topping Off"to prevent overflow.

Aircraft

- Tanker trucks (aviation fueling vehicles) should be equipped with spill response kits.
- Periodically inspect valves on mobile aircraft fuelers and on aircraft wing tips.

Contingency Response

- Conduct proper spill reporting to the appropriate regulatory agencies.
- Maintain adequate supplies of spill response equipment and materials in accessible locations near areas where spills may likely occur. Furnish all maintenance vehicles with adequate supplies of spill response materials and appropriate spill response procedures.
- Develop and implement a Spill Prevention Control and Countermeasure Plan (SPCC), required in 40 CFR 112.3(a), (b).
- Conduct proper spill reporting to the appropriate regulatory agencies.

Inspection and Testing

- Record all maintenance activities and inspections relating to fueling equipment and containers in a log book.
- Underground fuel storage tanks should be tested as required by federal and state laws.
- Provide the appropriate level of employee training in the following areas; spill response and prevention, stormwater
  pollution prevention education, right-to-know awareness training, and hazardous materials management.
- Develop regular maintenance and inspection programs for oil/water separators.
- Characterize wastes collected from oil/water separators. Dispose of these wastes properly and provide appropriate employee training.

APPROACH TO FUTURE FACILITY DESIGNS AND UPGRADES:

- Design fueling areas to prevent the run-on of stormwater and the runoff of spills by employing the following approaches (where authorized by the Town of Islip):

  - Cover the fueling area if possible.
  - Use a perimeter drain or slope the fueling area to a dead-end sump or oil/water separator.
  - Pave the fueling area with concrete rather than asphalt.
  - Where covering is infeasible and the fuel dispensing area has an asphalt pavement surface, consider applying a suitable sealant that protects the asphalt

from spilled fuels.
- If a dead-end sump is not used to collect spills, install an appropriately sized oil/water separator.

APPROACH TO SPILLS AT EXISTING FACILITIES

- Prevent spills from entering storm drains, floor drains, other receiving waters, and soil, through use of absorbent pigs, booms, or other means.
- Minimize immediate fire and safety hazards.
- Report any discharge of petroleum greater than 5 gallons to NYSDEC within 2 hours of discovery. Notify NYSDEC and/or the National Response Center for releases of a reportable quantity of a hazardous substance.
- If appropriate, sound alarms.
- Remove and dispose contaminated soil/material.
- Remove and recover free product in surface or groundwaters.
- Repair or replace leaking equipment.
- Perform other actions which regulatory agencies may require.
- Install Stage I and Stage II vapor recovery systems on gasoline dispensing equipment.
- Install shear valves on all fuel dispensing equipment.
- Design facilities to include secondary containment where required and/or appropriate.
- Upgrade or replace existing fuel storage tanks to have leak detection, spill containment, and overfill prevention before December 22, 1998.

RELEVANT RULES AND REGULATIONS:

- 6 NYCRR 608, 611 Oil Spill Prevention and Compensation Act
  6 NYCRR 595-597 Chemical Bulk Storage
- 6 NYCRR Part 360 Solid Waste Regulations
- 6 NYCRR 360-14 Waste Oil Regulations
- 6 NYCRR Parts 612-614 Petroleum Bulk Storage
- 6 NYCRR Part 371 Hazardous Waste Regulations
- 29 CFR 1910 (Subparts G, H, I, J, and K,) Hazardous Materials, Environmental Controls, and Personnel. Protection.
- 29 CFR 1910.1200 OSHA Hazard Communication Standard
- 40 CFR 122-124 NPDES Regulations for Stormwater Disrbarges
- 40 CFR 260-262, 268, and 270-272 Hazardous Waste Management
- 40 CFR 264-265 Preparedness, Prevention and Contingency Plan (PPCP)
- 40 CFR 302 Designation, Reportable Quantities and Notification Requirements for Hazardous Substances under CERCLA
- 40 CFR 372 Toxic Chemical Release Reporting: Community Right-to-Know
- 40 CFR 413, 433, and 469 NPDES Toxic Organic Management Plan (NPDESTOMP)
- 40 CFR 761 Toxic Substances
- 40 CFR 33 Oil Pollution Act of 1990
- 40 CFR 110.3 Discharge of Oil
- 40 CFR 112 Oil Pollution Prevention (SPCCIOPA Plans)
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous Substance
- 40 CFR 401 Effluent Limitation Guidelines

Town of Islip
Long Island MacArthur Airport

BMP3 AIRCRAFT, VEHICLE AND EQUIPMENT MAINTENANCE

PURPOSE:

Prevent or reduce the discharge of pollutants to surface water, groundwater, plus soil and air from aircraft, vehicle, and equipment maintenance and repair, including ground vehicle and floor washdowns.

APPROACH TO EXISTING FACILITY ACTIVITIES:

Operational Considerations

Minimum maintenance activities, including preflight checks and emergency repairs, shall employ the following to the extent practicable. All other maintenance shall be performed indoors or at off site facilities.

Good Housekeeping

- Use drip pans.
- Use absorbent materials at potential problem' areas. Collect/remove absorbent materials from area after use and dispose of them in an appropriate manner.
- Drain and crush oil filters (and oil containers) before recycling or disposal. Store crushed oil filters and empty lubricant containers in a leak-proof container - covered if outdoors.
- Label storm drain inlets to indicate they are to receive no wastes. Do not hose down work areas to the storm or sanitary drainage system or use concrete cleaning products unless the storm drain inlet is blocked and wash water is collected and properly disposed of through a permitted sewer connection. As an alternative, use mops, dry sweeping compound, or contract professional cleaning services. Confirm the use of appropriate disposal practices by contract cleaning services.
- Drain and properly dispose of all fluids and remove batteries from salvage aircraft, vehicles, and equipment.
- Recycle or properly dispose of the following: greases, oils, antifreeze, brake fluid, solvents or other cleaning solutions, hydraulic fluid, batteries, transmission fluid, and filters.
- Use biodegradable products and substitute materials with less hazardous properties where feasible.
- Tenants are required to use drip pans when performing maintenance such as adding oil or changing oil at the ramps.
- Tenants are educated on the potential impacts and risks of pollutants entering the drainage system as a result of maintenance activities.

Maintenance

- Maintain clean equipment by eliminating excessive amounts of external oil and grease buildup. Use water-based cleaning agents or non-chlorinated solvents to clean equipment.
- Regularly clean any oil water separators that receive wastewater from a maintenance area. Do not flush wastes into receiving waters. Activities such as painting and stripping, battery charging, and welding may require air permitting; check with local and state agencies for applicability.

Physical Site Usage

- Conduct maintenance activities indoors, or conduct off airport where possible.
- Use designated washing, steam cleaning, and degreasing areas to clean equipment.
- Store mechanical parts and equipment that may yield even small amounts of contaminants (i.e., oil or grease) under cover and away from drains.

Structural Control

- Maintenance and cleaning areas should be equipped with runoff controls that prevent discharge to storm and sanitary sewers.

Contingency Response

- Maintain adequate supplies of spill response equipment and materials in accessible locations near areas where spills may be likely to occur. Furnish all maintenance vehicles with adequate supplies of spill response materials and appropriate spill response procedures.

Inspection and Testing

- Provide the appropriate level of employee training in the following areas: spill response and prevention, stormwater pollution prevention education, right-to-know awareness training, and hazardous materials management.
- Provide employee stormwater quality awareness training.
- Develop regular maintenance and inspection programs for oil/water separators.
- Characterize wastes collected from oil/water separators. Dispose of these wastes properly and provide appropriate employee training.

APPROACH TO FUTURE FACILITY DESIGNS AND UPGRADES:

- Provide indoor maintenance areas when designing new facilities or upgrading existing facilities.
- Do not install floor drains in areas where maintenance is to be performed.

APPROACH TO SPILLS AT EXISTING FACILITIES

- Prevent spills from entering storm drains, floor drains, other receiving waters, and soil, through use of absorbent pigs, booms, or other means.
- Minimize immediate fire and safety hazards.
- If appropriate, sound alarms.
- Report any discharge of petroleum greater than 5 gallons to NYSDEC within 2 hours of discovery. Notify NYSDEC and/or the National Response Center for releases of a reportable quantity of a hazardous substance.
- Remove and dispose contaminated soil/material.
- Remove and recover free product in surface or groundwater.
- Repair or replace leaking equipment.
- Perform other actions which regulatory agencies may require

RELEVANT RULES AND REGULATIONS:

- 6 NYCRR 608, 611 Oil Spill Prevention and Compensation Act
- 6 NYCRR 595-597 Chemical Bulk Storage Regulations
- 6 NYCRR Part 360 Solid Waste Regulations
- 6 NYCRR 360-14 Waste Oil Regulations
- 6 NYCRR Parts 612-614 Petroleum Bulk Storage Regulations
- 6 NYCRR Part 371 Hazardous Waste Regulations
- 29 CFR 1910 (Subparts G, H, I, J, and K,) Hazardous Materials, Environmental Controls, and Personnel Protection.
- 29 CFR 1910.1200 OSHA Hazard Communication Standard
- 40 CFR 122-124 NPDES Regulations for Stormwater Discharges
- 40 CFR 260-262, 268, and 270-272 Hazardous Waste Management
- 40 CFR 264-265 Preparedness, Prevention and Contingency Plan (PPCP)
- 40 CFR 302 Designation, Reportable Quantities and Notification Requirements for Hazardous Substances under CERCLA
- 40 CFR 372 Toxic Chemical Release Reporting: Community Right-to-Know
- 40 CFR 413, 433, and 469 NPDES Toxic Organic Management Plan (NPDESTOMP)
- 40 CFR 761 Toxic Substances
- 40 CFR 33 Oil Pollution Act of 1990

Town of Islip
Long Island MacArthur Airport

BMP4 AIRCRAFT, VEHICLE AND EQUIPMENT WASHING, STEAM CLEANING AND DEGREASING

PURPOSE:

Prevent or reduce the discharge of pollutants to soil, groundwater, and stormwater drains from aircraft, vehicle, and equipment washing, and equipment degreasing.

APPROACH TO EXISTING FACILITY ACTIVITIES:

Aircraft, vehicle, and equipment washing, steam cleaning, and degreasing are prohibited at ISP with the following exceptions:

- Vehicles, aircraft, and equipment are washed indoors only and any wastewater generated must be disposed of off site by a licensed hauler.
- Washing is prohibited at ISP's outdoor areas as there are no sanitary sewer connections at the airport.
- Collection of wash water is not an acceptable alternative. For cleaning activities allowed at ISP, refer to the following operational, contingency and training approaches.

Operational Considerations

Implement the following to the maximum extent practical:

Good Housekeeping

- Use "dry" washing and surface preparation techniques where feasible. Several products are available to clean even the largest aircraft. Remove all materials (i.e., drippings and residue) using vacuum methods. Dispose of properly.
- Provide secondary containment for containers of washing and steam cleaning additives.
- Use biodegradable phosphate-free detergents.
- Keep washing area clean and free of waste.
- Include proper signage indicating that the discharge of waste oils into the drains is strictly prohibited.
- Discharge wash water to an approved treatment facility (sanitary sewer system) through a permitted connection.
- Ensure that wash water discharged to the sanitary sewer meets the pretreatment standards of the local POTW which treats the water.

Physical Site Usage

- Consider off site commercial washing and steam cleaning where feasible. Using appropriate off-site facilities that will decrease the waste generated onsite.
- Cover catch basins
- Provide training
- Do not contract with auto dealers or other cleaning companies that use wet

operations discharging to the storm drain system or receiving waters.

- Always use facility-designated indoor wash areas, or the ISP designated outdoor wash area, to prevent contamination of stormwater by contact with wastes.

Maintenance

- Inspect, clean, and maintain oil/water separators that are connected to indoor floor drains and receive discharges of washwater.

Contingency Response

- Maintain adequate supplies of spill response equipment and materials in accessible locations near areas where spills may be likely to occur.

Inspection and Training

- Provide the appropriate level of employee training in the following areas: spill response and prevention, stormwater pollution prevention education, right-to-know awareness training, and hazardous materials management.
- Develop regular maintenance and inspection programs for oil/water separators.
- Characterize wastes derived from oil/water separators.

APPROACH TO FUTURE FACILITY DESIGNS AND UPGRADES:

- Consider off-site commercial washing where feasible. Using appropriate off site facilities that will decrease the waste generated on-site.

APPROACH TO SPILLS AT EXISTING FACILITIES

- Prevent spills from entering storm drains, floor drains, other receiving waters, and soil, through use of absorbent pigs, booms, or other means.
- Minimize immediate fire and safety hazards.
- Report any discharge of petroleum greater than 5 gallons to NYSDEC within 2 hours of discovery. Notify NYSDEC and/or the National Response Center for releases of a reportable quantity of a hazardous substance (see Spills Management).
- If appropriate, sound alarms.
- Remove and dispose contaminated soil/material.
- Remove and recover free product in surface waters or groundwaters.
- Repair or replace leaking equipment.
- Perform other actions which regulatory agencies may require.

RELEVANT RULES AND REGULATIONS:

- 6 NYCRR Part 360 Solid Waste Regulations
- 6 NYCRR Parts 370-373 Hazardous Waste Regulations
- New York State Environmental Conservation Law
- 40 CFR 112 Oil Pollution Prevention for Spill Prevention Control and Countermeasure (SPCC) Plan
- 40 CFR 110.3 Discharge of Oil
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous

Substance

- 40 CFR 122-124 NPDES Regulations for Stormwater Discharges
- 40 CFR 401 Effluent Limitation Guidelines

Town of Islip
Long Island MacArthur Airport

BMP5 BUILDING CLEANING AND MAINTENANCE

PURPOSE:

Comply with state and federal regulations regarding safe work practices for equipment which may be used in building cleaning and maintenance. Prevent or reduce the discharge of pollutants to soil or groundwater via storm sewer. Sources of pollutant discharges include equipment blowdown, waste handling and disposal, and other discharges. Prevent air emissions from building maintenance, cleaning and HVAC operations.

APPROACH TO EXISTING FACILITY ACTIVITIES:

Operational Considerations

Pesticides

- Develop integrated pest management programs where appropriate.
- Minimize use of pesticides and fertilizers. Use pesticides and fertilizers according to
  directions. Seek less harmful/toxic pesticides and fertilizers to replace ones currently used.
- Use only certified pesticides applicators.
- Have pesticide product information available at facility; make information available to tenants and employees.
- Maintain a schedule for pesticide application and a record of pesticide usage.
- When performing self-application,
  - make sure employees are certified pesticide applicators
  - have effective backflow prevention devices for application equipment - provide proper personnel protection equipment to persons handling,
  loading, mixing, and applying pesticides
  - store pesticides in their original containers with legible labels. - send annual report to NYSDEC detailing pesticide application.

Stationary Combustion Installations

- Perform annual boiler inspections and tune-ups.
- Operate boilers and other stationary combustion installations within their permissible limits and maintain a log of boiler operation and maintenance.
- If possible, do not allow discharges of boiler blowdown to the sanitary sewer and never discharge blowdown to the storm sewer system. Properly dispose blowdown with other oily wastewaters.
- Check that fuel composition meets NYSDEC requirements for sulfur.

RELEVANT RULES AND REGULATIONS:

- 6 NYCRR Part 368 Recycling Emblems
- 6 NYCRR Parts 325 and 326 Pesticide Storage, Handling, and Disposal
- NYS Environmental Conservation Law, Article 33
- 6 NYCRR Parts 370-374 New York State Hazardous Waste Management Regulations
- 6 NYCRR Part 360 New York State Solid Waste Disposal Regulations
- 6 NYCRR Part 376 New York State Land Disposal Restrictions
- 29 CFR 1910 Subpart N-Material Handling and Storage
- 29 CFR 1910.1200 OSHA Hazard Communication Standard
- 40 CFR 112 Oil Pollution Prevention for Spill Prevention and Control and Countermeasure (SPCC) Plans
- 40 CFR 122 National Pollutant Discharge Elimination System (NPDES) Regulation
- 40 CFR 260-262, 268, and 270-272 Hazardous Waste Management
- 40 CFR 302 Designation, Reportable Quantities and Notification Requirements for Hazardous Substances under Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
- 40 CFR 372 Toxic Chemical Release Reporting: Community Right to Know
- 40 CFR 761 Toxic Substances
- 40 171-173, 175 and 177 Department of Transportation Regulations
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous Substance
- 40 CFR 122-124 NPDES Regulations for Stormwater Discharge
- 40 CFR 401 Effluent Limitation Guidelines

Town of Islip
Long Island MacArthur Airport

BMP6 CHEMICAL AND PETROLEUM STORAGE AND HANDLING

PURPOSE:

Ensure compliance with state and federal regulations regarding registration, handling, and storage of chemicals. Prevent or reduce discharge of pollutants to storm sewer or air by minimizing storage of materials on site, storing materials in designated areas, installing secondary containment and conducting regular inspections of storage areas. Storage of chemicals and petroleum products can pose the following risks: stormwater pollution, injury to workers or visitors, groundwater pollution, soil contamination, air pollution.

APPROACH TO STORAGE AT EXISTING FACILITIES:

Operational Considerations

Monitoring

- Perform inventory monitoring of all chemicals delivered to and stored on site; report inventories on annual basis, whenever required by state or local agencies.
- Maintain inventories in as small a quantity as practical.
- Perform weekly inspection of tanks and containers for leaks.

Storage

- Designate areas for chemical storage of containers (may not be possible for tanks).
- Store all chemical and petroleum products in a covered area.
- Store containers within secondary containment, such as within berms or dikes.
- Keep all chemicals in original containers.
- Keep all containers closed when not in use to prevent spills and air emissions.
- Maintain legible labels on all containers and tanks. Label all fillports of petroleum tanks with symbols and colors that are consistent with the American Petroleum Institute Standards.
- Comply with fire codes for storage of ignitable, flammable, or reactive liquids.
- To the extent practical, minimize storage and handling.

Training

- Train employees in storage procedures.
- Train employees in spill prevention and clean-up procedures.

Compliance

- Comply with all applicable state and federal regulations applying to tanks including requirements for:

- registration of aboveground storage tanks (ASTs) and underground storage tanks (USTs) inspection of storage tanks
- preparation of Spill Prevention Report for facilities with chemical storage tanks
- preparation of Spill Prevention Control and Countermeasure Plan for aboveground petroleum storage greater than 600 gallons in one container or 1320 gallons total, or for facilities with more than 40,000 gallons of underground storage
- labeling
- inventory monitoring and associated record keeping
- UST tightness testing, AST inspections and associated record keeping tank closure
- Major On-Shore Storage Facilities (>400,000 gallons on-site petroleum storage) must maintain sufficient records to substantiate average daily throughput and quantity of monthly fuel transfers.
- pay monthly license fee
- When taking tanks temporarily out-of-service, follow state and federal standards for temporary closure. When placing tanks permanently out-of-service, follow state and federal standards for permanent closure; report change in service to NYSDEC.

APPROACH TO SPILLS AT EXISTING FACILITIES:

- Prevent spills from entering storms drains, floor drains, other receiving waters, and soil, through use of absorbent pigs, booms, or other means.
- Minimize immediate fire and safety hazards.
- Report any discharge of petroleum greater than 5 gallons to NYSDEC within 2 hours of discovery. Notify NYSDEC and/or the National Response Center for releases at or above the reportable quantity of hazardous substance.
- If appropriate, sound alarms.
- Remove and dispose of contaminated soil/material.
- Remove and recover free product in surface or ground waters.
- Repair or replace leaking equipment.
- Perform other actions which regulatory agencies may require.

Contingency Response

- Develop and implement a Spill Prevention Control and Countermeasure (SPCC) Plan, if required under guidelines set forth in 40 CFR, Section 112.3(a), (b).
- Develop and implement a Spill Prevention Report (SPR), if required under the guidelines set forth in 6 NYCRR Parts 595-599.
- Maintain adequate supplies of spill response equipment and materials in accessible locations near areas where spill maybe likely to occur.

Inspection and Training

- Inspect containers frequently for leaks and proper closure seal.
- Develop employee training programs which emphasize the proper storage and handling procedures for chemical and petroleum products.
- Provide the appropriate level of employee training in the following areas: spill response and prevention, storm water pollution prevention education, right-to-know awareness training, and hazardous materials management.

APPROACH TO FUTURE FACILITY DESIGNS AND UPGRADES:

- Existing underground chemical and petroleum storage tanks should be upgraded with leak detection, spill containment, and overfill protection before December 22, 1998, the federal regulatory deadline.
- Incorporate adequate chemical and petroleum storage facilities into future buildings.
- Design facilities which have sheltered (covered) material storage areas.
- Construct secondary containment in proposed aboveground chemical and petroleum storage areas.
- Do not construct floor drains in areas where chemicals will be stored.
- When determining the size the layout of the chemical storage room, consider fire codes for reactive, flammable, and ignitable materials.

RELEVANT RULES AND REGULATIONS:

- 6 NYCRR Parts 595-599 Chemical Bulk Storage Regulations
- 6 NYCRR Parts 610, 612-614 Petroleum Bulk Storage Regulations
- 6 NYCRR Parts 608, 611 Oil Spill Prevention and Compensation Act
- New York State Environmental Conservation Law Articles 37 & 40
- New York State Uniform Fire Prevention and Building Code
- 40 CFR 110.3 Discharge of Oil
- 40 CFR 112 Oil Pollution Prevention (SPCC/OPA Plans)
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous Substance
- 40 CFR 122-124 NPDES Regulations for Stormwater Discharges
- 40 CFR 401 Effluent Limitation Guidelines
- 40 CFR 302 Designation, Reportable Quantities and Notification Requirements for Hazardous Substances under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA).
- 40 CFR 280-281 Underground Storage Tanks
- 40 CFR 372 Chemical Release Reporting: Community Right-to-Know
- 29 CFR 1910.1200 OSHA Hazard Communication Standard
- 49 CFR 171-173, 175 and 177 Department of Transportation Regulations

Town of Islip
Long Island MacArthur Airport

BMP7 ELIMINATION OF NON-STORM WATER DISCHARGES TO STORM DRAINS

PURPOSE:

Existing discharges: Eliminate non-stormwater discharges to the stormwater collection system. Non-stormwater discharges can be classified as follows: washwater, deicing fluids, and spillage, process wastewater, treated cooling water, and sanitary wastewater (through a pipe).

Prevention of illicit connections: Physical connections to the storm drain system from sanitary sewers, floor drains, industrial process discharge lines, and wash racks that are not expressly permitted by local, New York State or Federal governing agencies.

APPROACH TO EXISTING FACILITY ACTIVITIES:

General

- Perform frequent activity inspections to identify non-stormwater discharges - stagger inspection times to cover all work periods.
- Perform visual inspections of discharge points to the storm drain system - observe uncharacteristic volumes, colors, turbidity, odors, deposition, staining, floatables, and foaming characteristics of any flow.

Operational Considerations

- Use dry cleaning and surface preparation techniques where feasible.
- Limit the availability of outdoor water supplies (hose bibs).
- Post signs at outdoor water sources stating the appropriate uses and discouraging uses which would introduce pollutants to the storm drain system/receiving waters.
- Mark storm drains for easy identification and prevention of illicit discharges

Required Plan and/or Permits

- Owners and operators of facilities that store, process, or refine oil or oil products may be required by federal law (40 CFR 112) to develop and implement a Spill Prevention Control and Countermeasure (SPCC) Plan. In addition, owners or operators of a hazardous bulk storage facility (defined by NYSDEC Chemical Bulk Storage Regulations) are required to prepare and maintain a Spill Prevention Report (SPR). See BMP-8, "Spills Management," for additional information.

Contingency Response

- Follow spill notification and reporting procedures as described in BMP-8, "Spills Management." Follow contingency plans for spill containment as described in facility's SPCC or SPR, if applicable.
- Maintain adequate supplies of spill response equipment and materials in accessible locations near areas where spill may be likely to occur.
- Inspect waste containers frequently for leaks and proper closure seals. Keep dumpsters covered and plug any holes to prevent leaks from waste materials or run-through of liquid wastes and/or rainwater.
- Develop employee training programs which emphasize the proper storage and disposal procedures for operations derived wastes, particularly waste waters. (See BMP 13, "Waste Management")
- Provide the appropriate level of employee training in the following areas: spill response and prevention, stormwater pollution prevention education, right-to-know awareness training, and hazardous materials management.

APPROACH TO FUTURE FACILITY DESIGNS AND UPGRADES:

- Provide marking on storm drains for easy identification and prevention of illicit discharges.
- Perform inspections during the design review and project construction phases to ensure drainage, wastewater, and water supply connections are correct (no cross connections or illicit hookups).
- Develop a set of as-built drawings for all projects. Keep a set of the drawings at the facility.
- Design projects to include adequate waste repositories at locations near waste origin points.
- Provide adequate and appropriately designed facilities for functions such as painting, mechanical maintenance, chemical/fuel storage and delivery, material handling, waste handling and storage, lavatory service, and food preparation.

APPROACH TO SPILLS AT EXISTING FACILITIES:

- Prevent spills from entering storm drains, floor drains, other receiving waters, and soil, through use of absorbent pigs, booms, or other means.
- Minimize immediate fire and safety hazards.
- Report any discharge of petroleum greater than 5 gallons to NYSDEC within 2 hours of discovery. Notify NYSDEC and/or the National Response Center for releases of a reportable quantity of a hazardous substance
- If appropriate, sound alarms.
- Remove and dispose contaminated soil/material.
- Remove and recover free product in surface or ground waters.
- Repair or replace leaking equipment.
- Perform other actions which regulatory agencies may require.

RELEVANT RULES AND REGULATIONS:

- 6 NYCRR Parts 612-614 Petroleum Bulk Storage Regulations
- 6 NYCRR Parts 595-599 Chemical Bulk Storage Regulations
- New York State Environmental Conservation Law, Title 8, Article 17
- 40 CFR 110.3 Discharge of Oil
- 40 CFR 112 Oil Pollution Prevention (SPCC/OPA Plans)
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous Substance
- 40 CFR 122-124 NPDES Regulations for Stormwater Discharges
- 40 CFR 401 Effluent Limitation Guidelines

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BMP8 SPILLS MANAGEMENT

PURPOSE:

Prevent or reduce the discharge of pollutants to soil, groundwater, stormwater, surface water or air resulting from spills. Develop spill prevention plans to contain accidental and continuous releases of petroleum products or hazardous substances. Identify proper reporting procedures to implement in the event of a spill.

APPROACH TO EXISTING FACILITY ACTIVITIES:

Operational Considerations

- Post a summary of the plan at appropriate site locations, identifying the spill cleanup coordinators, location of cleanup equipment, and phone numbers of regulatory agencies to be contacted in the event of a spill.
- Maintain an inventory of appropriate cleanup materials on-site and strategically deploy cleanup materials based on the type and quantities of chemicals present.
- Make absorbents readily available in fueling areas.
- A spill clean-up contractor should be under contract with the tenant and available immediately during emergency situations.

Required Plans

Owners and operators of facilities that store, process, or refine oil or oil products maybe required by federal law (40 CFR 112) to develop and implement a Spill Prevention Control and Countermeasure (SPCC) Plan. In addition, owners or operators of a hazardous bulk storage facility (defined by NYSDEC Chemical Bulk Storage Regulations) are required to prepare and maintain a Spill Prevention Report (SPR). SPCC and SPR plans should include the following information:

A description of the facility including the owner's name and address, the nature of the facility activity, and the general types and quantities of chemicals stored at the facility.

- A site plan showing the location of storage areas for chemicals, the location of storm drains, site drainage patterns, fire water source locations, and the location and description of any devices used to contain spills such as positive shut-off control valves.
- Notification procedures to be implemented in the event of a spill, such as key company personnel and local, state, and federal agencies.
- Instructions regarding cleanup procedures.
- Designated personnel with overall spill response cleanup responsibility.

Spill prevention plans should be periodically updated as physical changes are made to the facility (e.g. layout, number of tanks, types of processes).

Notification Requirements

- Notify all applicable local, state and federal agencies in the event of a spill, including the following:

  - Local Fire Department : 911
  - NYSDEC Spills Hotline : 1-800-457-7362
  - National Response Center - if spill exceeds the reportable quantity (RQ): 1-800-424-8802 - NYCDEP Spill Hotline: 1-718-595-4646
  - US Coast Guard - if spill is near or has potential to enter navigable waters of the US including surface waters and adjoining shorelines: 1-718-354-4109

- Most spills of petroleum and hazardous substances (above the RQ) must be reported within two hours.

- Provide relevant information regarding the spill including:

  - Time, date, location, duration and estimated quantity of the release
  - Source of the release and identification of substance released into the environment
  - Individuals with authority/responsibility to implement or oversee clean-up

Contingency Response

- Implement spill containment and cleanup procedures immediately, as described in SPCC Plan and/or SPR.
- Maintain contract with spill clean-up contractor for immediate mobilization during emergency situations.
- Contact clean-up contractors as identified in spill prevention plans.
- Perform follow-up reporting procedures as required by regulatory agencies or as identified in the facility's SPCC Plan and/or SPR.
- For continuous releases (e.g., ethylene glycol), provide required telephone and written notifications to appropriate state and local agencies.
- Properly dispose any materials that have been contaminated as a result of a spill.

Inspection and Training

- Provide appropriate training for key personnel, with additional training for first responder level personnel (29 CFR 1910.120). All employees should have basic knowledge of spill control procedures.

APPROACH TO FUTURE FACILITY DESIGNS AND UPGRADES:

- Design bulk storage facilities which utilize effective spill prevention and containment technology.
- Locate bulk storage facilities in areas which minimize potential discharge to soil, groundwater, surface water, storm water or sanitary systems.

RELEVANT RULES AND REGULATIONS:

- 40 CFR 110.3 Discharge of Oil
- 40 CFR 112 Oil Pollution Prevention (SPCC/OPA Plan)
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous Substance
- 40 CFR 122-124 NPDES Regulations for Storm Water Discharges
- Article 12, New York State Navigation Law
- 6 NYCRR Parts 612-614 Petroleum Bulk Storage Regulations
- 6 NYCRR Parts 595-599 Chemical Bulk Storage Regulations
- 6 NYCRR Parts 608, 611 Oil Spill Prevention and Compensation Act
- 40 CFR 372 Toxic Chemical Release Reporting: Community Rights to Know
- 40 CFR 302 Resignation, Reportable Quantities and Notification Requirements for Hazardous Substance under CERCLA

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BMP 9 OIL WATER SEPARATORS

PURPOSE:

Oil/water separators are baffled chambers designed to remove petroleum compounds and greases from storm water. Oil/water separators also remove floatable debris and settled solids (sediment).

APPROACH TO EXISTING FACILITY ACTIVITIES:

Operational Considerations

- Separators must be inspected and cleaned frequently of accumulated oil, grease, floating debris and sediments to be effective storm water quality controls.
- Oil absorbent pads are to be replaced as needed but should always be replaced prior to the wet season.
- The effluent shutoff valve should be closed during cleaning operations.
- Any standing water removed during the cleaning operation must be disposed of in accordance with federal, state, and local requirements.
- Any standing water removed during the cleaning operation must be replaced with clean water to prevent oil carry-over through the outlet.

Contingency Response

- Maintain adequate supplies of spill response equipment and materials in accessible locations near areas where spills may be likely to occur.

Inspection and Training

- Provide the appropriate level of employee training in the following areas: spill response and prevention, storm water pollution prevention education, right-to-know awareness training, and hazardous materials management.
- Perform and document in a log book all inspections and maintenance operations.
- Develop a written operating, sampling, and reporting procedure under local storm water authority guidelines.
- Train appropriate employees to implement these procedures.

APPROACH TO FUTURE FACILITY DESIGNS AND UPGRADES:

Design of New Facilities and Existing Facility Upgrades:

Oil/water separators are typically used in areas where the concentrations of petroleum hydrocarbons, floatables, or sediment may be abnormally high and source control techniques are not very effective. There are two types of oil/water separators: the American Petroleum Institute (API) separator and the coalescing plate separator (CPS). Design, sizing, and placement of oil/water separators is dependent on several factors

including: tributary area, type of activity, pollutant type and concentration, and water temperature. General sizing guidelines for API separators include the following:

- Horizontal velocity: 3 feet per minute.
- Depth of 3 to 8 feet.
- Depth-to-width ratio of 0.3 to 0.5.
- Width of 6 to 16 feet.
- Baffle height-to-depth ratios of 0.85 for top baffles and 0.15 for bottom baffles.

CPS separator sizing is more complex. Sizing calculations require the inclusion of information such as packing plate surface areas and plate angles. CPS separators can, due to their packed plate design, remove the same quantities of oils and greases while occupying less space than API separators.

RELEVANT RULES AND REGULATIONS:

- 6 NYCRR Parts 608, 611 Oil Spill Prevention on Compensation Act
- 6 NYCRR Part 360-14 Waste Oil Regulations
- 6 NYCRR Parts 612-614 Petroleum Bulk Storage Regulations
- 40 CFR 40 Effluent Limitation Guidelines
- 40 CFR 110.3 Discharge of Oil
- 40 CFR 112 Oil Pollution Prevention (SPCC/OPA Plans)
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous Substance
- 40 CFR 122-124 NPDES Regulations for Stormwater Discharges

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BMP 10 OUTDOOR HANDLING OF MATERIAL

PURPOSE:

Prevent or reduce the discharge of pollutants to soil, groundwater, surface water or stormwater from loading and unloading of material and cargo.

APPROACH TO EXISTING FACILITY ACTIVITIES:

Operational Considerations

Good Housekeeping

- Use seals or door skirts between vehicles and structures to prevent material exposure to rainfall.
- Contain and adsorb leaks during transfers and spillage from hose disconnections; dispose of residue properly.
- Avoid transferring materials in close proximity to storm drain inlets.
- Use drip pans under hoses.
- Transfer liquids only in paved areas. Portland cement paving should be used if the liquid is asphalt reactive.
- Provide contractors and haulers with copies of pertinent GEPs. Require contractor/hauler adherence to GEP specifications.
- Consider contracting maintenance operations for material handling equipment. Designate an appropriate area for contractors to perform maintenance activities. Verify proper waste disposal practices of contractors.

Physical Site Usage

- Protect all loading/unloading activities from rainfall, run-on and wind dispersal to the maximum extent practical. Viable options include conducting loading/unloading under existing cover, or moving indoors.
- Position tank trucks or delivery vehicles so that possible spills or leaks can be contained.

Structural Controls

- Cover loading/unloading areas/docks to reduce exposure of materials to rain. Construct roofing structure over material handling area, or move indoors.

Maintenance

- Conduct berm repair and patching.
- Inspect, clean and maintain oil/water separators.

Contingency Response

- Maintain adequate supplies of spill response equipment and materials in accessible locations near areas where spills may be likely to occur.
- Include spill kits on appropriate material handling vehicles and equipment.

Inspection and Training

- Conduct regular inspections and make repairs as necessary. Document inspections.
- Check loading/unloading equipment (valves, pumps, flanges, and connections) regularly for leaks. Document inspections.
- Develop and implement a written operations plan which describes loading/unloading procedures.
- Provide proper training for material handling equipment operators. Include periodic refresher training.
- Provide the appropriate level of employee training in the following areas: spill response and prevention, stormwater
  pollution prevention education, right-to-know awareness training, and hazardous materials management. Include periodic
  refresher training.

APPROACH TO FUTURE FACILITY DESIGNS AND UPGRADES:

- Design loading/unloading areas to prevent stormwater run-on through the use of the following practices:
  - Grading or berming.
  - Positioning roof downspout to direct stormwater away from loading/ unloading areas.
- Design facilities so that materials which may contribute pollutants to stormwater may be stored indoors or under cover.
- Incorporate oil/water separators into exposed loading dock designs.

APPROACH TO SPILLS AT EXISTING FACILITIES:

- Prevent spills from entering storm drains, floor drains, other receiving waters, and soil, through use of absorbent pigs, booms, or other means.
- Minimize immediate fire and safety hazards. If appropriate, sound alarms.
- Report any discharge of petroleum greater than 5 gallons to NYSDEC within 2 hours of discovery. Notify NYSDEC and/or the National Response Center for releases of a reportable quantity of a hazardous substance.
- Remove and dispose contaminated soil/ material.
- Remove and recover free product in surface or ground waters.
- Repair or replace leaking equipment.
- Perform other actions which regulatory agencies may require.

RELEVANT RULES AND REGULATIONS:

- 6 NYCRR Parts 6085611 Oil Spill Prevention and Compensation Act
- 6 NYCRR Parts 612-614 Petroleum Bulk Storage Regulations
- 6 NYCRR Parts 595-599 Chemical Bulk Storage Regulations
- 40 CFR 110.3 Discharge of Oil
- 40 CFR 112 Oil Pollution Prevention (SPCC/OPA Plans)
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous Substance
- 40 CFR 122-124 NPDES Regulations for Stormwater Discharges

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BMP 11 OUTDOOR MATERIAL AND EQUIPMENT STORAGE

PURPOSE:

Prevent or reduce the discharge of pollutants to soil, groundwater, surface water, or stormwater from outdoor storage areas for significant material (e.g., fuels, chemicals, bagged material on pallets, soils or asphalt material bulk storage, deicing compounds, and equipment etc.).

APPROACH TO EXISTING FACILITY ACTIVITIES:

Operational Considerations

Good Housekeeping

- Avoid dispensing from drums positioned horizontally in cradles. Dispense materials from upright drums equipped with hand pumps if possible. Always use drip pans and self-closing spigots, if dispensing from horizontally positioned drums.
- Store drums and containers on pallets or other structures to keep the container out of contact with stormwater.
- Use drum lids to prevent rainfall from washing materials and drippage from the top of containers to the storm drain system.
- Discharge collected storm water from secondary containment areas according to guidelines developed by the federal government and applicable state and local regulations.
- Store all materials in their original containers or containers approved for that use.
- Ensure that all containers are appropriately sealed. Store empty containers indoors or under cover or move them off site.
- Properly label all chemical containers with information, including their contents, hazards, spill response and First-aid procedures, manufacturer's name and address, and storage requirements. See local and New York State requirements for labeling. Maintain copies of MSDS on file for any materials stored and/or handled by the applicator.

Physical Site Usage

- Protect all significant materials from rainfall, run-on, runoff and wind dispersal to the maximum extent practical. Viable options are: - Store material indoors.
  - Cover the storage area with a roof.
  - Cover the material with a temporary covering made of polyethylene, polypropylene, or hypalon.
  - Minimize stormwater run-on by enclosing the area, building a berm around the area, store indoors, or completely cover.

- Reduce the quantities of significant materials stored outside (i.e., chemicals) to the minimum volume required based on variables such as release potential, usage, and shelf life.
- Make use of existing overhangs to the extent practicable.

Structural Controls

- Provide berming or secondarily contain storage tankers, ASTs, drums and containers.
- Install, maintain, and replace catch basin filter inserts consistent with manufacturers specifications.

Maintenance

- Inspect, clean and maintain sumps, on a regular basis.
- Periodically inspect all equipment stored outdoors, including trash compactors, vehicles, etc. If leaks are noted, repair immediately. If repairs are not possible, confine and contain the leak, ensuring that it does not enter any storm drains until clean up or reports can be completed.

Contingency Response

- Develop and implement a Spill Prevention Control and Countermeasures (SPCC) Plan, if required under guidelines set forth in 40 CFR, Section 112.3(a), and (b), submit to NYSDEC for approach.
- Maintain adequate supplies of spill response equipment and materials in accessible locations near areas where spills may be likely to occur.
- Post appropriate signs at all chemical storage locations in clearly visible locations noting the materials stored, emergency contacts, and spill cleanup procedures.

Inspection and Training

- Perform and document periodic inspections in a logbook. Inspection items should include the following:
  - External corrosion and structural failure.
  - Spills and overfills due to operator failure.
  - Failure of piping system (pipes, pumps, flanges, couplings, hoses, and valves).
  - Leaks or spills during pumping of liquids or gases.
  - Loose fittings, poor welds, and improper or poorly fitted gaskets.
  - Tank foundations and storage area coatings.

- Provide the appropriate level of employee training in spill response and prevention, stormwater pollution prevention, right-to-know awareness, and hazardous material management.

RELEVANT RULES AND REGULATIONS

- 40 CFR 110.3 Discharge of Oil
- 40 CFR 112 Oil Pollution Prevention (SPCC/OPA Plans)
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous Substance
- 40 CFR 122-124 NPDES Regulations for Stormwater Discharges
- 40 CFR 401 Effluent Limitation Guidelines
- 6 NYCRR Parts 595-599 Chemical Bulk Storage Regulations
- 6 NYCRR Parts 612-614 Petroleum Bulk Storage Regulations
- 6 NYCRR Part 360 Solid Waste Disposal Regulations
- 6 NYCRR Parts 370-374 New York State Hazardous Waste Management

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BMP 12 WASTE MANAGEMENT

PURPOSE:

Minimize waste production and increase awareness in waste management options. Prevent or reduce discharge of pollutants to soil, groundwater, surface water and stormwater. Comply with state and federal regulations pertaining to the generation and disposal of solid and hazardous waste.

APPROACH TO EXISTING FACILITY ACTIVITIES:

General

In general, the following is an appropriate hierarchy for waste generation and disposal:
REDUCE = REUSE =RECYCLE = DISPOSE

Reduce: Evaluate areas where operations can be altered to minimize the production of waste. Examples: a) Amount of wastewater generated during vehicle washing can be minimized by installation of a washwater recovery and recycling system; b) At times, the amount of packaging can be minimized.

Reuse: Some materials can be reused several times before disposal. Examples:
a) Packing material can often be reused; b) Installation of a solvent recovery system will allow solvent to be used several times before being Aspent.n Recycle: Identify operations that generate cardboard, wood pallets, used oil, metals, plastic and glass. If possible, source separate and send to appropriate recycling facility.

Dispose: This is the last option for material that no longer appears to have any reuse capabilities and is a material that cannot be recycled. This material (e.g. food waste, aircraft cabin waste) should be sent to a licensed disposal facility.

In order to apply the waste management hierarchy, identify all waste streams, including aircraft waste and note the most common method of disposition of each waste type. Consider developing a waste minimization plan for the facility based on the waste management hierarchy. The plan should define strategies for waste minimization based on the analysis of facility waste streams. The plan should be made available to all employees.

Operational Considerations

Tracking

- Characterize waste streams, and evaluate the process generating the waste. Do not mix wastes that have not been characterized as hazardous or nonhazardous.
- Determine whether waste is considered to be hazardous or non-hazardous. This determination can be made by aknowledge of the process generating the waste and the type of waste material (ignitable, reactive, corrosive, RCRA listed waste, etc.) or by chemical analysis.

- Avoid mixing hazardous and non-hazardous wastes to minimize the quantity that must be disposed in accordance with hazardous waste regulations.

Wastewater Discharges

- Clean any catch basins that receive stormwater runoff from maintenance areas on a regular basis. Use a vacuum truck to remove accumulated materials. Do not flush any wastes into receiving waters.
- Prevent leaks from dumpsters and compactors from entering storm drains.
- For equipment that does not blowdown continuously, collect blowdown in a container and properly dispose with other oily waste waters; do not dispose into storm drain or floor drains.
- Avoid discharges of wastewater into floor drains, by sealing floor drains in areas were industrial wastewater is likely to be discharged.
- Ensure that equipment that blows down continuously is not discharging to a storm drain.
- Clearly mark storm drains for easy identification and prevention of illicit discharges.

Miscellaneous

- Maintain a minimal inventory of required chemicals to reduce the magnitude of potential spills and limit waste generation.
- Find substitutes for harmful chemicals - properly dispose of unusable chemical inventory.
- Encourage employees to recommend areas where operations can be altered to minimize waste generation.

Contingency Response

- Maintain adequate supplies of spill response equipment and materials in accessible locations near areas where spills may be likely to occur.
- Equip waste transport vehicles with spill containment equipment.
- Collect outdoor washdown water and properly dispose of it through a permitted connection to an approved treatment facility. Approval from treatment facility required for discharge.
- Report all spills to the appropriate regulatory agencies.

Inspection and Training

- Provide the appropriate level of employee training in the following areas; spill response and prevention, stormwater pollution prevention education, right-to-know awareness training, waste minimization techniques, and hazardous materials management. Include periodic refresher training.
- Perform and document in a log book periodic inspections of hazardous and non-hazardous waste storage areas.

Inspection items should include the following:

- Check for external corrosion and structural failure.
- Check for spills and overfills due to operator failure.

- Check for failure of piping system (pipes, pumps, flanges, couplings, hoses, and valves).

APPROACH TO FUTURE FACILITY DESIGNS AND UPGRADES:

- Perform inspections during the design review and project construction phases to ensure drainage, wastewater, and water supply connections are correct (no cross connections or illicit hookups).

- Develop a set of as-built drawings for all projects. Keep a set of the drawings at the facility.

- Design projects to include adequate waste storage areas at locations near waste generation points.

- Provide adequate and appropriately designed facilities for functions such as steam cleaning, degreasing, painting, mechanical maintenance, chemical/fuel storage and delivery, material handling, waste handling and storage, lavatory service, and food preparation.

RELEVANT RULES AND REGULATIONS:

- 6 NYCRR Part 368 Recycling Emblems
- 6 NYCRR Parts 325 and 326 Pesticide Storage, Handling, and Disposal
- NYS Environmental Conservation Law, Article 33
- 6 NYCRR Parts 370-374 New York State Hazardous Waste Management Regulations
- 6 NYCRR Part 360 New York State Solid Waste Disposal Regulations
- 6 NYCRR Part 376 New York State Land Disposal Restrictions
- 29 CFR 1910 (Subparts G, H, I, J and K) Hazardous Materials, Environmental Controls, and Personnel Protection
- 29 CFR 1910 Subpart N- Material handling and Storage
- 29 CFR 1910.1200 OSHA Hazard Communication Standard
- 40 CFR 112 Oil Pollution Prevention for Spill Prevention and Control and Countermeasure (SPCC) Plans
- 40 CFR 122 National Pollutant Discharge Elimination System (NPDES) Regulation
- 40 CFR 260-262, 268, and 270-272 Hazardous Waste Management
- 40 CFR 302 Designation, Reportable Quantities and Notification Requirements for Hazardous Substances
  under Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
- 40 CFR 372 Toxic Chemical Release Reporting: Community Right to Know
- 40 CFR 761 Toxic Substances
- 40 CFR 171-173, 175 and 177 Department of Transportation Regulations
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous Substance
- 40 CFR 122-124 NPDES Regulations for Stormwater Discharges
- 40 CFR 401 Effluent Limitation Guidelines

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BMP 13 STORM WATER POLLUTION PREVENTION EDUCATION

PURPOSE:

Prevent or reduce the discharge of pollutants to storm water from activities through implementing an education program targeting employees, vendors, and the public.

APPROACH TO FUTURE FACILITIES AND UPGRADES:

Design of New Facilities and Existing Facility Upgrades

- Work early on with design and construction engineers and local storm water authorities to incorporate proactive storm water management features into projects such as decreased impervious areas, infiltration BMPs, biofilters, oil/water separators, etc.

- Inform all construction contractors of their responsibility to comply with adopted BMPs and with regulations prohibiting cross connections between sanitary sewers and storm drains. Provide contractors and subcontractors with copies of relevant BMPs during specification and bidding phases.

APPROACH TO EXISTING FACILITY ACTIVITIES:

Contingency Response

- Provide adequate implementation training for facilities with a Spill Prevention Control and Countermeasure (SPCC) Plan, if required developed under guidelines set forth in 40 CFR, Section 112.3(a), (b).

- Adequately train employees in the use of spill response equipment and materials.

Inspection and Training

- Perform and document in a log book frequent inspections of work areas, waste storage facilities, maintenance areas, and contractor projects to examine compliance with BMPs. Follow up with additional training or enforcement as required. Incorporate inspection findings into subsequent training efforts.

- Design storm water pollution education programs to contain the following elements:

  - Promote the proper storage, use, and disposal of landscape maintenance chemicals and other potentially harmful chemicals.
  - Promote the use of safer alternative products such as: short-lived pesticides, non-chlorinated solvents, water-based paints, non-aerosol products. Encourage the use of "dry" washing processes for aircraft, vehicles, and equipment.

- Design storm water pollution education programs to contain the following

elements:

-Encourage efficient and safe housekeeping practices in industrial activity areas.
- Increase awareness of the detrimental environmental impacts that result when fuel, antifreeze, pesticides, lubricants, detergents, paints and other wastes are dumped onto the ground or into storm drains.
- Promote source reduction and recycling of waste materials.
- Increase awareness of possible penalties and fines associated with discharge of pollutants into storm drains.
- Increase awareness of what is and what is not allowed to enter storm drains.
- Provide a mechanism for violations to be reported.

REQUIREMENTS:

- Capital and O&M costs are minimal for educational programs.
- Educational programs need to be ongoing. Information and training must be disseminated at regular intervals.

LIMITATIONS:

- The success of educational programs is difficult to measure. Acceptance and awareness are critical factors.

RELEVANT RULES AND REGULATIONS:

- 40 CFR 110.3 Discharge of Oil
- 40 CFR 112 Oil Pollution Prevention (SPCC/OPA Plans)
- 40 CFR 117.3 Determination of Reportable Quantities for a Hazardous Substance
- 40 CFR 122-124 NPDES Regulations for Storm Water Discharges
- 40 CFR 401 Effluent Limitation Guidelines

Town of Islip
Long Island MacArthur Airport

BMP 14 PAVEMENT SWEEPING & STORM WATER FACILITY MAINTENANCE

PURPOSE:

The solids and floatable control aspect of this BMP focuses on using preventive measures to reduce the amount of solids and floatable materials entering the storm system. Many solids on the street come from pavement, tire and vehicle equipment wear, and often contain heavy metals and petroleum products. Solids are also produce by erosion along roads.

APPROACH TO EXISTING FACILITY ACTIVITIES:

Pavement sweeping

All aeronautical areas and roadways will be swept on an as required basis. Airport employees will inspect the stability of shoulders, embankments, ditches and soils along the streets at least twice a year. Eroding sites will be repaired.

Stormwater Facility Maintenance

The Airport is required to maintain all stormwater facilities to ensure that they are properly functioning and operating at the designed efficiency. A catch basin is a vault or a chamber that is usually associated with the storm drain inlet. The catch basin usually has the capability to trap debris and some sediment before they travel farther into the storm system. If catch basins are not cleaned periodically, they may fill up with debris and stop functioning properly. To mitigate that possibility, the Airport schedules annual catch basin cleaning, where all sediment and debris from pavement runoff -s removed and the vault is cleaned out. At the same time the basin is inspected for proper operation.

RELEVANT RULES AND REGULATIONS:

- NYS Environmental Conservation Law, Article 33
- 40 CFR 112 Oil Pollution Prevention for Spill Prevention and Control and Countermeasure (SPCC) Plans
- 40 CFR 122 National Pollutant Discharge Elimination System (NPDES) Regulation
- 40 CFR 171-173, 175 and 177 Department of Transportation Regulations
- 40 CFR 122-124 NPDES Regulations for Stormwater Discharges
- 40 CFR 401 Effluent Limitation Guidelines

Town of Islip
Long Island MacArthur Airport

BMP 15 SECURITY

PURPOSE:

Heightened security began before the new Aviation Security and Transportation Act of 2001 became law on November 19, 2001. Criminal background checks on all airport employees have begun and must be completed by year-end. Carry-on bags are searched more carefully, and passengers' names are crossed-checked with lists of people the Federal government deems suspicious.

In addition, the Airport recently announced additional implementation of airport security measures that will require all current and prospective employees who have access to secure areas of airports to undergo fingerprinting and criminal history background checks.

The Airport also will make greater use of closed circuit television cameras to supplement access control systems to help law enforcement personnel determine the nature of an intrusion or an alarm.

Appendix E

Annual Site Compliance Evaluation

Annual Site Compliance Evaluation
Long Island MacArthur Airport (ISP)
Ronkonkoma, NY 11779

Th Annual Site Compliance Evaluation includes procedures and protocols for inspections to be performed to adhere to specific Best Management Practices (BMPs) identified in the BMPP required by the New York State Department of Environmental Conservation (NYSDEC) State Pollutant Discharge Elimination System (SPDES) Permit for ISP. TOI shall use this evaluation form to assess BMPP implementation effectiveness and follow-up actions .

I. FACILITY INFORMATION

Instructions: Give the tenant/facility's name, terminal, and building numbers, and address or location of tenant's facility. Give the tenant contact name, as it is legally referred to, of the person, business, public organization, or other entity that owns or operates the tenant's facility. Give the name, title, and work telephone number of the Pollution Prevention Team (PPT) member who is thoroughly familiar with the operation of the facility and with the facts reported in this BMPP Implementation Inspection Report and who can be contacted by the TOI for additional information.

1. TENANT/FACILITY NAME	**2.** TERMINAL NUMBER(s)	**3.** BUILDING NUMBER(s)	
4. MAILING ADDRESS/LOCATION OF PROPERTY	**5.** CITY	**6.** STATE	**7.** ZIP CODE
8. TENANT CONTACT (Name)	**9.** MAILING ADDRESS (if different from No. 4.)	**10.** CONTACT PHONE NUMBER	
11. POLLUTION PREVENTION TEAM MEMBER (Name)	**12.** CONTACT PHONE NUMBER	**13.** EMAIL ADDRESS	
14. COMMENTS	**15.** NOTES		

Nature of Facility

Instructions: Provide a brief description of the nature of the business in the space provided.

16. PROPERTY TYPE (Circle One): COMMERCIAL TELECOMMUNICATIONS PARKING CARGO MAINTENANCE OTHER (Specify) _____

17. VENDORS (Circle One): Yes or No _____

18. PROVIDE A GENERAL DESCRIPTION OF THE FACILITY OPERATIONS _____

_____

_____

_____

Additional Remarks:

_____

_____

II. BMPP IMPLEMENTATION INSPECTION

Instructions: Before proceeding to conduct the BMPP Inspection and complete the BMPP Implementation Inspection Form please verify that a review of the facility components and systems has been completed to identify potential industrial activities that contribute to stormwater pollution discharges at ISP located in Appendix D of the ISP BMPP.

19. HAS THE MEMBER WHO IS THOROUGHLY FAMILIAR WITH THE OPERATION OF THE FACILITY REVIEWED THE ISP BMPP? (Circle One) **YES NO**

20. DO TENANT PERSONNEL RECEIVE GENERAL TRAINING FOR MANAGING OF RUNOFF FROM SITE? (Circle One) **YES NO**

21. DO TENANT PERSONNEL RECEIVE SPECIFIC TRAINING FOR PREVENTING POLLUTION AND CONTROLLING RUNOFF FROM SITE (BMP IMPLEMENTATION)? (Circle One) **YES NO**

22. ARE TRAINING RECORDS AND EDUCATIONAL MATERIALS AVAILABLE FOR REVIEW? (Circle One) **YES NO**

23. DOES FACILITY HAVE CURRENT ENVIRONMENTAL PLANS (Circle One) **YES NO** (Circle Applicable):
SPILL RESPONSE PLAN (SRP) SPILL PREVENTION CONTROL & COUNTERMEASURE PLAN (SPCC) OTHER (Specify) _____

BMPP IMPLEMENTATION INSPECTION FORM

Instructions: Check boxes to indicate if identified BMPs are being implemented for the following activities conducted at ISP. Please also check the box which best describes the effectiveness of BMP implementation as described below:

Check "SATISFACTORY" if BMPs (including source control BMPs) are used and are effective.

Check "NEEDS IMPROVEMENT" if either no BMPs are used, or if some BMPs are used but are not effective in minimizing non-stormwater discharges.

ISP BMPs	BMP Used			BMP Implementation Effectiveness	
	Yes	No	N/A	Satisfactory	Needs Improvement
BMP 1 AIRCRAFT DEICING.					
1. Is propylene glycol used at the facility? (If no, then proceed to next BMP, if yes, then complete remaining questions)	☐	☐	☐	☐	☐
2. Are appropriate glycol/water blends utilized during deicing?	☐	☐	☐	☐	☐
3. Are there plans to upgrade the existing truck fleet/spray nozzles?	☐	☐	☐	☐	☐
4. Does your facility have designated personnel that dispense the deicing material?	☐	☐	☐	☐	☐
5. Are personnel educated and trained regarding the use of deicing material?	☐	☐	☐	☐	☐
6. Is the type and quantity of deicing material used reported to the TOI monthly on the Deicing/Anti-icing Reporting Form?	☐	☐	☐	☐	☐
7. Are all propylene glycol (ethylene glycol is prohibited) releases reported to the appropriate entity and the TOI?	☐	☐	☐	☐	☐
BMP 2 AIRCRAFT, VEHICLE, AND EQUIPMENT FUELING.					
1. Are berms or curbing installed around fueling areas?	☐	☐	☐	☐	☐
2. Are absorbent materials and/or vacuum equipment available for spills?	☐	☐	☐	☐	☐
3. Are vapor recovery systems installed and utilized at fuel dispensers and tanks?	☐	☐	☐	☐	☐
4. Are canopies installed?	☐	☐	☐	☐	☐
5. Is preventive maintenance utilized to inspect, and correct? Malfunctions?	☐	☐	☐	☐	☐

ISP BMPs	BMP Used			BMP Implementation Effectiveness	
	Yes	No	N/A	Satisfactory	Needs Improvement
BMP 3 AIRCRAFT, VEHICLE, AND EQUIPMENT MAINTENANCE.					
1. Is maintenance performed indoors?	☐	☐	☐	☐	☐
2. Are floor drains located in maintenance areas?	☐	☐	☐	☐	☐
3. Are materials used in repair and minor remodeling (paints, etc.) stored properly?	☐	☐	☐	☐	☐
4. Are employees trained on spill prevention and response?	☐	☐	☐	☐	☐
5. Are safer alternative products used whenever possible?	☐	☐	☐	☐	☐
6. Are adequate records kept in accordance with applicable regulations?	☐	☐	☐	☐	☐
BMP 4 AIRCRAFT, VEHICLE, AND EQUIPMENT WASHING, STEAM CLEANING AND DEGREASING.					
1. Are dry washing techniques utilized in designated areas and wherever possible?	☐	☐	☐	☐	☐
2. Is wash water recycled or discharged appropriately?	☐	☐	☐	☐	☐
3. Are catch basins covered during washing operations?	☐	☐	☐	☐	☐
BMP 5 BUILDING CLEANING AND MAINTENANCE.					
1. Are pesticides and fertilizers used and stored properly?	☐	☐	☐	☐	☐
2. Is maintenance performed indoors?	☐	☐	☐	☐	☐
3. Are floor drains located in maintenance areas?	☐	☐	☐	☐	☐
4. Are materials used in repair and minor remodeling (paints, etc.) stored properly?	☐	☐	☐	☐	☐
5. Are employees trained on spill prevention and response?	☐	☐	☐	☐	☐
6. Are safer alternative products used whenever possible?	☐	☐	☐	☐	☐
7. Are adequate records kept in accordance with applicable regulations?	☐	☐	☐	☐	☐
BMP 6 CHEMICAL AND PETROLEUM BULK STORAGE HANDLING.					
1. Are routine inspections and maintenance performed at all locations?	☐	☐	☐	☐	☐
2. Are employees trained on spill prevention and response?	☐	☐	☐	☐	☐
3. Are all spills reported to appropriate regulatory agencies and the TOI ?	☐	☐	☐	☐	☐
4. Are adequate records kept in accordance with applicable regulations?	☐	☐	☐	☐	☐
BMP 7 ELIMINATION OF NON-STORMWATER DISCHARGES TO STORM DRAINS.					
1. Are inspections and enforcement with regular corrective action measures conducted at the facility?	☐	☐	☐	☐	☐
2. Are employees trained on methods to eliminate discharges to storm drains?	☐	☐	☐	☐	☐
3. Has the BMPP been distributed to all and reviewed by all appropriate staff, tenant personnel/contractors?	☐	☐	☐	☐	☐
BMP 8 SPILLS MANAGEMENT.					
1. Does the facility maintain and implement a SPCC?	☐	☐	☐	☐	☐
2. Is immediate containment/cleanup of spills performed?	☐	☐	☐	☐	☐
3. Are applicable absorbent materials and vacuum cleaners immediately accessible for spill events?	☐	☐	☐	☐	☐
4. Are all spill events reported to appropriate regulatory agencies and the TOI ?	☐	☐	☐	☐	☐

ISP Airport BMPs	BMP Used			BMP Implementation Effectiveness	
	Yes	No	N/A	Satisfactory	Needs Improvement
BMP 9 OIL/WATER SEPARATORS.					
1. Are oil/water separators inspected and cleaned frequently of accumulated oil, grease, floating debris, and sediment?	☐	☐	☐	☐	☐
2. Are absorbent pads replaced on a routine basis?	☐	☐	☐	☐	☐
3. Is standing water removed during cleaning operations?	☐	☐	☐	☐	☐
BMP 10 OUTDOOR HANDLING OF MATERIAL.					
1. Is loading/unloading conducted under cover?	☐	☐	☐	☐	☐
2. Is transferring material next to storm drain inlets avoided to the maximum extent possible?	☐	☐	☐	☐	☐
3. Are drip pans used under hoses?	☐	☐	☐	☐	☐
4. Are material transfer leaks immediately contained and absorbed?	☐	☐	☐	☐	☐
BMP 11 OUTDOOR MATERIAL AND EQUIPMENT STORAGE.					
1. Are covers used to protect all raw materials, by-products, finished products items stored outside?	☐	☐	☐	☐	☐
2. Are chemicals, drums, or bagged materials on pallets or similar method that keep them off the ground?	☐	☐	☐	☐	☐
3. Are hazardous materials (if permitted in lease) stored in properly designed secondary containment areas?	☐	☐	☐	☐	☐
4. Are drip pans and containers used in areas where drips or leaks may occur?	☐	☐	☐	☐	☐
5. Are berms, curbs, or other structures in place to minimize pollutants from entering the storm water system?	☐	☐	☐	☐	☐
6. Are all containers properly labeled?	☐	☐	☐	☐	☐
7. Are periodic inspections conducted for storage areas?	☐	☐	☐	☐	☐
BMP 12 WASTE MANAGEMENT.					
1. Are measures taken to streamline operations to minimize waste?	☐	☐	☐	☐	☐
2. Are wastes segregated/separated/recycled to maximum extent?	☐	☐	☐	☐	☐
3. Is storage area designed to prevent storm water runoff?	☐	☐	☐	☐	☐
4. Are waste dumpsters covered?	☐	☐	☐	☐	☐
5. Are records of waste generation and disposal maintained?	☐	☐	☐	☐	☐
6. Are licensed firms utilized for waste disposal?	☐	☐	☐	☐	☐

ISP BMPs	BMP Used			BMP Implementation Effectiveness	
	Yes	No	N/A	Satisfactory	Needs Improvement
BMP 13 STORMWATER POLLUTION PREVENTION EDUCATION.					
1. Is an organization-wide BMP implementation training program in place to inform personnel?	☐	☐	☐	☐	☐
2. Does the program adequately explain components and goals of Activity Specific BMPs?	☐	☐	☐	☐	☐
3. Are mandatory and regular training sessions conducted for employees?	☐	☐	☐	☐	☐
4. Are training records kept in accordance with applicable regulations?	☐	☐	☐	☐	☐
BMP 14 PAVEMENT SWEEPING AND STORMWATER FACILITY MAINTENANCE.					
1. Is landside street sweeping conducted daily from Spring-Fall?	☐	☐	☐	☐	☐
2. Are aeronautical areas and roadways swept once per week?	☐	☐	☐	☐	☐
3. Is street sweeping performed as necessary during winter months?	☐	☐	☐	☐	☐
4. Are roadways inspected and repaired routinely?	☐	☐	☐	☐	☐
BMP 15 SECURITY.					
1. Are appropriate security measures taken to prevent unauthorized access at secure areas to prevent illicit discharges?	☐	☐	☐	☐	☐

III. BMP ASSESSMENT AND CORRECTIVE ACTION

Instructions: Visual inspections evaluate the effectiveness of stormwater BMPs. Provide any observations taken during the inspection that may warrant revisions to the BMPs. Specify any deficiencies in the BMPs inspected and note any corrective action that may/should be taken.

24. OBSERVATIONS/COMMENTS _____

_____

_____

25. CORRECTIVE ACTION _____

_____

_____

_____

_____

_____

IV. INSPECTION INFORMATION

Instructions: Provide name of party who conducted the inspection.

26. INSPECTOR NAME (Printed) _____

27. CONTACT PHONE NUMBER _____

28. DATE/TIME OF INSPECTION _____

29. TIME OF INSPECTION (Circle One) ANNUAL (DEICING SEASON – from November to April)

30. NAMES OF TENANTS ACCOMPANYING INSPECTOR _____

31. NAMES OF TENANTS ACCOMPANYING INSPECTOR _____

HELPFUL HINTS FOR STORMWATER INSPECTIONS

AREAS AND ACTIVITIES WITH POTENTIAL TO SPILL HAZARDOUS FLUIDS

- ✓ SPILL KITS AVAILABLE IF NEEDED
- ✓ SPILL RESPONSE AGENCY PHONE NUMBERS CLEARLY POSTED
- ✓ MATERIAL SAFETY DATA SHEETS AVAILABLE FOR PRODUCTS

PARKING LOTS

- ✓ PAVED AREAS SWEPT DOWN AND NOT HOSED OFF
- ✓ TRASH RECEPTACLES AVAILABLE TO PREVENT LITTER
- ✓ SPILLS OR LEAKS SPOT CLEANED AS NEEDED

BUILDING AND GROUNDS MAINTENANCE

- ✓ VEGETATION LEFT IN PLACE WHERE POSSIBLE
- ✓ SOIL EROSION NOT OBSERVED
- ✓ GROUNDS FREE OF LITTER
- ✓ PAVED AREAS SWEPT DOWN AND NOT HOSED OFF
- ✓ SAFER ALTERNATIVE PRODUCTS IN USE
- ✓ TRASH RECEPTACLES AVAILABLE TO PREVENT LITTER
- ✓ MINIMAL USE OF PESTICIDES AND FERTILIZERS

PARK AND LANDSCAPE MAINTENANCE

- ✓ SOIL EROSION NOT OBSERVED
- ✓ GROUNDS FREE OF LITTER
- ✓ PAVED AREAS SWEPT DOWN AND NOT HOSED OFF
- ✓ IRRIGATION SYSTEMS DESIGNED TO PREVENT RUNOFF
- ✓ USE OF PESTICIDES AND FERTILIZERS ONLY AS NEEDED

OUTDOOR LOADING/UNLOADING OF MATERIALS

- ✓ MATERIAL LOADING/UNLOADING AREAS ENCLOSED OR COVERED
- ✓ LOADING AND UNLOADING CONDUCTED IN DRY WEATHER IF NOT COVERED
- ✓ LOADING AND UNLOADING AREA BERMED OR SLOPED TO CONTAIN SPILLAGE
- ✓ NO DISCHARGE TO STORM DRAIN
- ✓ DRIP PANS AVAILABLE TO CAPTURE LIQUID LEAKS

OUTDOOR STORAGE OF MATERIALS/PRODUCTS/EQUIPMENT

- ✓ HAZARDOUS MATERIALS LABELED, COVERED, AND CONTAINED
- ✓ STOCKPILED MATERIALS COVERED
- ✓ NO SIGNS OF EXCESSIVE LEAKING FROM STORED EQUIPMENT
- ✓ DRIP PANS AVAILABLE TO CAPTURE EQUIPMENT LEAKS
- ✓ STORAGE AREA FREE OF LITTER
- ✓ GENERAL GOOD HOUSEKEEPING OBSERVED

WASTE HANDLING AND DISPOSAL

- ✓ RECYCLABLE MATERIALS BEING RECYCLED
- ✓ WASTE CONTAINERS COVERED TO PREVENT STORM WATER RUNOFF/RUN-ON
- ✓ DRIP PANS AVAILABLE TO CAPTURE GREASE WHEN TRANSFERRED
- ✓ AREA SWEPT DOWN ON REGULAR BASIS
- ✓ AREA FREE OF STAINS OR OTHER SIGNS OF POLLUTANTS GOING INTO STORM DRAIN SYSTEM
- ✓ WASTE CONTAINERS IN GOOD CONDITION FREE FROM LEAKS
- ✓ GENERAL GOOD HOUSEKEEPING OBSERVED
- ✓ NO LITTERING SIGNS POSTED

ERODIBLE SURFACE AREAS

- ✓ PRESERVE NATURAL VEGETATION
- ✓ VEGETATION LEFT IN PLACE WHERE POSSIBLE
- ✓ SOIL EROSION NOT OBSERVED
- ✓ CHEMICAL STABILIZATION OR GEOSYNTHETICS IN USE ON BARE GROUND
- ✓ PAVED AREAS SWEPT REGULARLY

ILLICIT CONNECTIONS/ILLEGAL DISCHARGES

- ✓ SIGNS OF ILLICIT CONNECTIONS TO STORM WATER CONVEYANCE SYSTEM(S)
- ✓ SIGNS OF ILLEGAL/UNKNOWN DISCHARGE OBSERVED GOING FROM THE LEASE PREMISES ONTO ADJACENT PROPERTY OR INTO THE STREET
- ✓ SIGNS OF ILLEGAL/OR UNKNOWN DISCHARGE OBSERVED COMING FROM ADJACENT PROPERTY ONTO THE LEASE PREMISES
- ✓ STAINED PAVEMENT IN AREAS NEAR OR SURROUNDING CATCH BASIN OR STORM WATER OUTFALL

BMPP IMPLEMENTATION INSPECTION FORM

- BMPP Implementation Effectiveness
- Results of BMP Inspection
- Presented in Annual Facility Review and Site Inspection Report
- Ensure Training of BMPP
- Completed Semi-Annually and at least during Deicing/Anti-icing Season

BMPP KEY COMPLIANCE

- Avoid costly permit violations and problems meeting SPDES permit compliance requirements with proper inspection and maintenance of BMPs.
- Stormwater BMPs require regular inspections and maintenance to ensure long-term proper function.
- Inspections are the most effective way to detect and avoid potential problems before they become a liability.

FOR ADDITIONAL INFORMATION CONTACT:

Al Cinotti
Chief Fire Safety Officer
Town of Islip – Department of Aviation
Long Island MacArthur Airport (ISP)
Bayport Aerodrome (23N)
100 Arrival Avenue, Suite 100
Ronkonkoma, NY 11779

631.467.3279

www.FlyMacArthur.com

Arcadis U.S., Inc.
17-17 Route 208 North, Suite 290 West
Fair Lawn
New Jersey 07410
Phone: 201 797 7400
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www.arcadis.com

Appendix F

Community Air Monitoring Plan (CAMP)

Town Of Islip

Community Air Monitoring Plan

Long Island MacArthur Airport
100 Arrival Avenue, Ronkonkoma, New York
NYSDEC Site # 152251

June 4, 2026

Community Air Monitoring Plan

Long Island MacArthur Airport

March 31, 2026

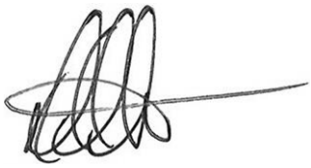
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Prepared For:
Town of Islip
Long Island MacArthur Airport[Title]
Ronkonkoma, New York

Our Ref:
30166065.RIWP2023



Kevin Held, CIH, CSP
HASP Reviewer



David Stern, LEP
Project Manager

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2 Monitoring Instrumentation 1
3 Monitoring Frequency 1
3.1 VOC Monitoring Stations Locations, Response Levels, and Action 2
3.2 Particulate Monitoring Stations Locations, Response Levels, and Actions 3

This document is intended only for the use of the individual or entity for which it was prepared and may contain information that is privileged, confidential and exempt from disclosure under applicable law. Any dissemination, distribution or copying of this document is strictly prohibited.

1 Introduction

In accordance with New York State Department of Health (NYSDOH) requirements, this Community Air Monitoring Plan (CAMP) has been prepared for use during remedial investigation activities performed at the Long Island MacArthur Airport (ISP or Site) located at 100 Arrival Avenue, Ronkonkoma, NY. This CAMP serves to present the methods and procedures to conduct real-time monitoring for volatile organic compounds (VOC) and total particulates (i.e., fugitive dust) at each designated work area when certain activities are in progress.

This CAMP is not intended for use in establishing action levels for worker respiratory protection; action levels are described in the Site-Specific Health and Safety Plan (HASP) (Appendix E of the RI-FS Work Plan). The intent of this CAMP is to provide a measure of protection for the downwind community (i.e., off-site receptors including residences and businesses and on-site workers that are not directly involved with the subject work activities) from potential airborne contaminant releases as a direct result of investigative activities that are related to the Site. The response levels specified herein require increased monitoring, corrective actions to abate emissions, and/or work shutdown. Additionally, this CAMP helps to confirm that work activities do not spread contamination off-site through the air.

Reliance on this CAMP does not preclude simple, common-sense measures to keep potential dust and odor emissions at a minimum around work areas. On the contrary, the air monitoring proposed is intended to enhance and record the effectiveness of good work practices in the control of potential emissions. The following sections of this CAMP present the monitoring instrumentation required to comply with NYSDOH policy, the frequency of monitoring, response levels, and response actions.

2 Monitoring Instrumentation

VOC monitoring will be performed using real-time monitoring instrumentation that can measure the types of VOCs known or suspected to be present at the work location (please refer to the HASP for details). The equipment will be calibrated daily and using the methods described in the HASP. Fifteen-minute running average concentrations will be recorded using photo-ionization detector (PID) MiniRAE 3000 or similar. The monitoring equipment will be with an audible alarm to indicate exceedance of the action level.

The particulate monitoring will be performed using real-time instrumentation that can measure total particulate. Fifteen-minute running average concentrations will be recorded using a dust monitor (i.e., TSI Dust Track II, ThermoFisher Scientific PDR 1000, or similar). The particulate monitoring equipment will have an audible alarm to indicate exceedance of the action level.

3 Monitoring Frequency

This section defines the typical activities that will occur in relation to the work area and correlates these activities to the frequency of monitoring required. VOC monitoring will only be conducted if working in an area where hydrocarbons or other VOCs could potentially be present. Particulate monitoring will be required when working in all areas of the site for intrusive activities listed below, not just in Area's of Concern identified in the Interim Site Management Plan.

Continuous Monitoring for VOC and Particulates During Intrusive Activities identified below:

1. Soil boring and sampling
2. Drilling for and installation of temporary and permanent monitoring wells
3. Construction activities involving earthwork or disturbance of earthen surfaces
4. Other activities specified in this CAMP

Additionally, upwind particulate concentrations will be measured at the **start** of each workday and **periodically** (see below) thereafter to establish the background concentration.

Periodic monitoring for VOCs will be carried out during non-intrusive activities if working in areas where VOCs could potentially be present. For non-intrusive activities, the upwind concentrations will be measured at the **start and finish** of the work effort to establish the background concentration. Non-intrusive activities typically include the following:

1. Site Mobilization/Demobilization of equipment and machinery
2. Surveying (geophysical, coordinate/elevation)
3. Waste transportation
4. Site preparation and restoration that does not involve re-grading or other disturbances to surface materials

"Periodic" monitoring should be performed, at a minimum as follows:

1. Upon arrival at a work location to determine the ambient, or background concentrations
2. During each phase of work that potentially may generate VOC emissions to the air
3. Prior to leaving the work location

As an example, "Periodic" monitoring for VOCs during sample collection activities shall include monitoring as above and during the following times:

1. When opening drums or containers, or overturning soil
2. During collection of samples (soil/sediment/groundwater)

For non-intrusive activities, particulate monitoring will not be performed.

3.1 VOC Monitoring Stations Locations, Response Levels, and Action

During each workday, the VOC monitoring station will be positioned at the downwind perimeter of the work area (i.e., the exclusion zone – see HASP for definition). As stated above, monitoring frequency (periodic or continuous) will be determined based on whether the activity is considered intrusive or non-intrusive. The direction of wind (if any) will be periodically recorded during each workday and re-positioning of upwind/downwind monitoring stations will be performed accordingly.

The VOC monitoring instrumentation output documenting 15-minute running average concentrations will be compared to the following response levels:

- If the ambient air concentration of total organic vapors at the downwind perimeter of the work area exceeds 5 parts per million (ppm) above background for the 15-minute average, work activities will be temporarily halted and monitoring continued.

If the total organic vapor level readily decreases (per instantaneous PID readings) below 5 ppm over background, work activities will resume with continued monitoring.

- If total organic vapor levels at the downwind perimeter of the work area or exclusion zone persist at levels in excess of 5 ppm above background but less than 25 ppm, work activities will be halted, the source of vapors identified, corrective actions taken to abate emissions, and monitoring continued. After these steps, work activities will resume provided that the total organic vapor level 200 feet downwind of the work area or half the distance to the nearest potential receptor or residential/commercial structure, whichever is less - but in no case less than 20 feet, is below 5 ppm over background for the 15- minute average.
- If the organic vapor level is above 25 ppm at the perimeter of the work area, activities will be shut down.

All readings will be recorded on the appropriate air monitoring log (please refer to the HASP for details) or the electronic log will be printed out. Air monitoring results will be appended to the appropriate report.

3.2 Particulate Monitoring Stations Locations, Response Levels, and Actions

For intrusive activities, the particulate (i.e., dust) monitoring station will be positioned at the downwind perimeter of the work zone (i.e., outside the work zone). In addition, fugitive dust migration will be visually assessed during all work activities. The direction of wind (if any) will be periodically recorded during each workday and re-positioning of the downwind monitoring station will be performed accordingly. The response levels and actions for fugitive dust are as follows:

- If the downwind PM-10 particulate level is 0.1 milligrams per cubic meter (mg/m3) greater than background (upwind perimeter) for the 15-minute period or if airborne dust is visually observed leaving the work area, then dust suppression techniques will be employed. Dust suppression techniques will include (but not be limited to) misting of soil excavation activities, wet cutting of asphalt or concrete as necessary, wetting of roadways and work areas. Work will continue with dust suppression techniques provided that downwind particulate levels do not exceed 0.1 mg/m3 above the upwind level and provided that no visible dust is observed leaving the work area.
- If, after implementation of dust suppression techniques, downwind particulate levels are greater than 0.1 mg/m3 above the background concentration, then work will be stopped, and a re-evaluation of activities initiated. Work will resume if dust suppression measures and/or other controls are successful in reducing the downwind particulate concentration to less than 0.1 mg/m3 of the upwind level and in preventing visible dust from leaving the work area.

All readings will be recorded on the appropriate air monitoring log or the electronic log will be printed out. Air monitoring results will be appended to the appropriate report

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