CUMMINGS
RITER
CONSULTANTS, INC.

REVISED WORK PLAN
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
KOPPERS POND
KENTUCKY AVENUE WELLFIELD SUPERFUND SITE
OPERABLE UNIT 4
HORSEHEADS, NEW YORK

PREPARED FOR:
KOPPERS POND RI/FS GROUP
HORSEHEADS, NEW YORK

PROJECT NO. 502.10/01
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REVISED WORK PLAN
REMEDIAL INVESTIGATION/FEASIBILITY STUDY
KOPPERS POND
KENTUCKY AVENUE WELLFIELD SUPERFUND SITE
OPERABLE UNIT 4
HORSEHEADS, NEW YORK

1.0 INTRODUCTION

The Koppers Pond RI/FS Group (the Group) has retained Cummings/Riter Consultants, Inc. (Cummings/Riter) to prepare this Remedial Investigation/Feasibility Study (RI/FS) Work Plan (Work Plan) for Koppers Pond in Horseheads, New York (the Site). AMEC Earth and Environmental, Inc. (AMEC) has assisted in developing this Work Plan, particularly as related to evaluations of potential human health and ecological risks and quality assurance.

Cummings/Riter and AMEC have prepared this Work Plan in accordance with the requirements of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or "Superfund"); the National Oil and Hazardous Substances Pollution Contingency Plan (NCP); and, more specifically, Paragraph 27a(2) of the Administrative Settlement Agreement and Order on Consent for Remedial Investigation/Feasibility Study (Index No. CERCLA-02-2006-2025) (Settlement Agreement) entered into between the Group and the U.S. Environmental Protection Agency (USEPA) on September 28, 2006.

The Group previously submitted an RI/FS Work Plan to USEPA on June 18, 2007. USEPA provided preliminary review comments on that plan, and representatives of the Group, USEPA, and other reviewers met on October 11, 2007 to discuss the comments and proposed revisions to the RI/FS Work Plan. In a letter dated October 18, 2007, USEPA formally requested that the Group revise the RI/FS Work Plan pursuant to the preliminary review comments and the meeting discussions. This revised RI/FS Work Plan addresses the issues raised in USEPA's review and the proposed resolution of those issues as discussed with USEPA and other reviewers on October 11, 2007.

1.1 BACKGROUND

Under the Settlement Agreement, Koppers Pond is being addressed as Operable Unit 4 of the Kentucky Avenue Wellfield Superfund Site, which is located within the Village of Horseheads and the Town of Horseheads in Chemung County, New York (Figure 1). The Kentucky Avenue Well is a municipal water supply well owned by the Elmira Water Board (EWB) that was used as part of the EWB system to furnish potable water to local communities. The Kentucky Avenue Well was closed in 1980 when it was found that the groundwater produced from this well contained trichloroethylene (TCE). USEPA subsequently identified the former Westinghouse Electric Corporation (Westinghouse) Horseheads plant site, which is located approximately one mile north-northwest of the Kentucky Avenue Well (Figures 1 and 2), as a likely source of TCE in local groundwater. In 1983, USEPA included the Kentucky Avenue Wellfield Site on the National Priorities List for response actions under CERCLA.

Beginning in the mid-1980s, several CERCLA response actions have been completed with respect to the Kentucky Avenue Wellfield Site:

- Operable Unit 1 involved the initial Site investigations, identification of potentially impacted private wells, and connection of the affected residents to the public water supply system.

- Operable Unit 2 included the supplemental investigation of the degree and extent of groundwater impacts, the installation of barrier wells and groundwater treatment system to intercept TCE-impacted groundwater at the downgradient limits of the former Westinghouse Horseheads plant site, and restoration of the Kentucky Avenue Well.

- Operable Unit 3 comprised the investigation and remediation of identified source areas at the former Westinghouse Horseheads plant site, the investigation of a waterway (i.e., the "Industrial Drainageway") that conveys surface water discharges from the former Westinghouse Horseheads plant site to Koppers Pond, and the remediation of the Industrial Drainageway.

The response actions specified under Operable Units 1 and 3 are completed. Operation, maintenance, and monitoring activities are continuing with respect to the barrier wells and attendant groundwater treatment system installed under Operable Unit 2. The RI for Koppers Pond is being conducted under Operable Unit 4.

1.2 SCOPE AND ORGANIZATION OF RI/FS WORK PLAN

The objective of the RI is to characterize environmental media at the Site sufficiently to allow for the evaluation of the need for remedial action and, if remedial action is deemed necessary, for the development and evaluation of remedial alternatives in the FS. The RI is to provide the necessary physical, chemical, and biological information pertaining to potential impacts to surface water and sediment in Koppers Pond and use these data to evaluate potential human health and ecological risks posed by chemicals of potential concern (COPCs) associated with these media. This Work Plan has been prepared in accordance with the Statement of Work provided as Appendix A to the Settlement Agreement and pertinent USEPA guidance, including *Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA* (EPA 540/G-89/004, October 1988).

In developing and negotiating the Settlement Agreement and the Statement of Work attached thereto, USEPA and the Group recognized that several pertinent studies of the Kentucky Avenue Wellfield Site have already been completed and that much is known about the Site. As a result, the scope of the envisioned RI/FS was tailored to meet the specific circumstances for Koppers Pond. As described in more detail in this RI/FS Work Plan, however, conditions in Koppers Pond are dynamic, and certain aspects and characteristics of the pond have likely changed since the time data were collected as part of prior studies. Accordingly, the RI for Koppers Pond is focused on collecting current information regarding surface water and sediment quality and comparing these data to the results of previous studies. Fish tissue sampling will also be conducted to provide current data.

Based on these evaluations, the RI will then assess the extent to which past studies, including previously completed risk assessments, present findings that are representative of the current conditions in Koppers Pond. Where previously collected data and studies are found to be representative of and applicable to current conditions, these past investigation results and assessments will be incorporated into the RI. Where past results are found to be unrepresentative of current conditions, however, or where studies are found lacking with respect to more-recent USEPA guidance or other technical factors (e.g., chemical toxicity data), the previously collected data will not be presented as representative, and risk assessments will be updated or redone.

Because the future direction of the RI and risk assessments will be determined only after data-collection activities are completed, the discussions of the human health and ecological risk assessments in this RI/FS Work Plan primarily focus on the content of expected deliverables and the approach to assembling those deliverables. This approach recognizes the iterative and step-wise nature of the data evaluation and risk assessment process.

Following this introductory section, this Work Plan is organized as follows:

- Section 2.0 presents a discussion of the Site background, including the environmental setting;
- Section 3.0 discusses the results of previous investigations;
- Section 4.0 presents the Preliminary Conceptual Site Model (PCSM);
- Section 5.0 discusses the objectives of the RI/FS, including data quality objectives (DQOs);
- Section 6.0 provides a description of the RI/FS tasks to be completed at the Site; and
- Section 7.0 presents the project schedule.

Appended to this Work Plan are the following:

- Appendix A - Field Sampling Plan (FSP) portion of the Sampling and Analysis Plan (SAP);
- Appendix B - Quality Assurance Project Plan (QAPP) portion of the SAP; and
- Appendix C - Site-Specific Health and Safety Plan (HASP).

These appended plans provide specific procedural guidelines as to how the tasks described in the Work Plan will be performed.

2.0 SITE DESCRIPTION AND SETTING

This section provides an overview of the physical characteristics of Koppers Pond and its environs. Included are descriptions of Site features, climate, surface water hydrology, hydrogeology, local land use, and ecological setting. This information was gleaned from the results of previous investigations of the Kentucky Avenue Wellfield Site, primarily the RI Report for Operable Unit 3 (Philip Environmental Services Corporation, March 1996) and more recent Site inspections and reconnaissance.

2.1 SITE FEATURES

Koppers Pond is a man-made, V-shaped pond located in the Village of Horseheads, New York (Figure 2). At the northern end of its western leg, the pond receives inflow from a surface watercourse known as the Industrial Drainageway. This drainageway receives much of its base flow from discharges originating at the former Westinghouse Horseheads plant site (Figure 2). The overflow from Koppers Pond discharges to two outlet streams located at the southern end of the pond, which combine to form the outlet channel.

2.1.1 Pond

Koppers Pond is a shallow, flow-through water body with typical water depths of approximately three to six feet. Because of the relatively flat topography, the open water area of the pond is highly dependent on the surface water elevation, and open water areas of approximately seven to more than nine acres have been reported in the various studies of this pond. At a pond surface water elevation of 887± feet above mean sea level (ft-msl), as reflected in Figures 2 and 3, the open water area of the pond covers about 8.0 acres. At this level, the volume of the pond is estimated to be on the order of 12 million gallons.

The origin of the pond is not well documented. It is situated in a previously low-lying, wet area that apparently began to fill with water with the onset of discharges from the former Westinghouse plant, which began operating in 1952, and industrial development on the south side of the area that began around 1953. Examination of the 1953 U.S. Department of the Interior, Geological Survey (USGS) map of the 7.5-minute

Horseheads topographic quadrangle (Figure 4) does not show the pond or industrial activity to the south, but shows the Industrial Drainageway flowing through a 20+ acre marshy area in the vicinity of the current pond location. The marshy area lies below approximate elevation 890 ft-msl. The 1969 USGS map of the same quadrangle shows the pond at its current location, but much larger (20± acres) and in a somewhat different configuration (Figure 4). In the 1969 map, an additional section of the pond is situated to the north within the current "V." This section of the pond was apparently filled after 1969; a 1991 closure investigation report for the Old Horseheads Landfill shows this area having been filled with construction and demolition debris (Fagan Engineers, March 1991). The reported limits of construction and demolition debris disposal at the Old Horseheads Landfill are shown on Figure 5. Also since 1969, the southern bank, including the pond outlet, appears to have been reworked with a second outlet added on the western side of the pond. Chemung County Sewer and Water Conservation District aerial photographs from 1977 and 1985 show Koppers Pond in its present configuration.

2.1.2 Industrial Drainageway

The Industrial Drainageway begins at a point approximately 2,300 feet to the north-northwest of Koppers Pond at the outlet of a 74-inch diameter underground pipe (the "Chemung Street Outfall"). The underground pipe, which is approximately 1,600 feet in length, conveys discharges from the former Westinghouse Horseheads plant site and local storm water runoff. From the Chemung Street Outfall, the Industrial Drainageway flows to the south-southeast, discharging into Koppers Pond.

The 1953 USGS map shows the Industrial Drainageway as an open waterway extending to the approximate northern boundary of the former Westinghouse Horseheads plant site. The underground piping was installed after 1953 (but before 1969), perhaps as part of the New York State Department of Transportation (NYSDOT) construction of the New York Route 17/14 interchange in this vicinity.

Throughout most of its current 2,300-foot length, the drainageway is approximately 7 to 10 feet wide and varies in depth from about 0.5 to 2 feet. At its southern end, the Industrial Drainageway widens out to approximately 100 feet as it enters Koppers Pond. In this area, the Industrial Drainageway flows slowly through emergent vegetation (e.g., cattails) and is approximately 0.5 foot deep. The area surrounding the southern portion of

the Industrial Drainageway and the northwest corner of Koppers Pond has little topographic relief, and changes inflows and pond water levels can significantly alter the size and shape of these water bodies.

2.1.3 Outlet Channel

The two outlet streams that flow from the southern end of Koppers Pond merge about 500 feet downstream. After merging, the single outlet channel flows past the Hardinge, Inc. (Hardinge) plant site, and then into Halderman Hollow Creek. From that point, the creek flows south and southeast through mixed industrial, commercial, and residential areas, discharging into Newtown Creek approximately three miles south of Koppers Pond. Newtown Creek is a first-order tributary to the Chemung River.

2.2 CLIMATE

Chemung County, New York is characterized by a temperate climate with mild summer and long, cold winters. The annual average temperature is 47 degrees Fahrenheit (°F). August is the warmest month with average high temperatures above 80°F, but summers are moderate and average just 4 or 5 days per year with a maximum temperature of 90°F or above. Winter temperatures from December through February average below 30°F.

The average annual precipitation in Chemung County is approximately 33.5 inches, including the water equivalent of the annual average of 45 inches of snowfall. Precipitation is relatively uniformly distributed throughout the year. As presented in the Operable Unit 3 RI Report (Philip Environmental Services Corporation, March 1996), various studies have shown annual average runoff in the range of 7 to 10 inches per year.

2.3 SURFACE WATER HYDROLOGY

The Industrial Drainageway receives much of its base flow from discharges originating from permitted outfalls at the former Westinghouse Horseheads plant site (Figure 2). Historically, such discharges included treated process waste waters, non-contact cooling water, and storm water runoff. Although flow rates varied over time, total flows from these sources historically averaged between 1,000 and 2,000 gallons per minute (gpm) or 2.2 to 4.4 cubic feet per second (cfs).

Some process water discharges continue from the ongoing manufacturing operations of the Cutler-Hammer Division of Eaton Corporation (Cutler-Hammer) located at the former Westinghouse Horseheads plant site, but current discharges to the Industrial Drainageway from the former Westinghouse Horseheads plant site are primarily storm water runoff from building roofs and the treated effluent from the barrier well treatment facility installed under Operable Unit 2. Previously, the barrier well treatment facility effluent had been routed to the manufacturing operations at the former Westinghouse Horseheads plant site as a source of process water, but with the decreased manufacturing activity and lower demand, the barrier well treatment system effluent is now largely discharged directly into the Industrial Drainageway. The base, non-storm flow now averages approximately 1,400 gpm (3.2 cfs) from the former Westinghouse Horseheads plant site discharges.

Other sources of flow to the Industrial Drainageway include local surface water runoff. The contributory watershed area draining to the Industrial Drainageway at the point it enters Koppers Pond is estimated to be 604 acres, 59 acres of which comprise the former Westinghouse Horseheads plant site. At assumed basin-wide runoff rates of 7 to 10 inches per year, surface water runoff to the pond, excluding runoff from the former Westinghouse Horseheads plant site, would be about 200 to 280 gpm (0.4 to 0.6 cfs) as an annual average. With the total average inflow of approximately 1,600 gpm, the 12-million gallon water volume in Koppers Pond turns over at a rate of once per every 5.2 days or about 70 times per year. The storm-flow hydrology has not been studied; peak storm flows in excess of 20,000 gpm (44.6 cfs) are suspected based on observed channel size and drainage area.

Koppers Pond and the streams draining it are classified as Class C fresh surface waters by the New York State Department of Environmental Conservation (NYSDEC). Class C waters are to be suitable for fish propagation and survival and for primary and secondary contact recreation, such as swimming and boating.

2.4 HYDROGEOLOGY

The regional and local hydrogeology has been extensively studied with respect to the Kentucky Avenue Wellfield Site, and groundwater recovery, treatment, and monitoring are continuing under Operable Unit 2.

Koppers Pond lies within a large glacial outwash valley oriented north to south and extending southward to the Chemung River. The uppermost water-bearing zone occurs in thick glacial outwash sand and gravel deposits that overlie low-permeability lacustrine clays, glacial till, and bedrock. Thin surface alluvial or fluvial deposits occur locally. The base of the sand and gravel unit ranges in depth from about 30 to more than 60 feet below the ground surface (ft-bgs) in the vicinity of Koppers Pond. In some locations, discontinuous silt and clay layers of varying thickness occur within the sand and gravel sequence.

Local groundwater flow is toward the south and southeast at a relatively flat gradient. The outwash sand and gravel deposits are permeable, with hydraulic conductivities typically in the range of 50 to more than 2,000 feet per day. Well yields in excess of 500 to 1,000 gpm are common.

The silty sediments within Koppers Pond are less permeable than the outwash sand and gravel, but Koppers Pond nonetheless communicates with local groundwater. Depending on the time of the year and antecedent rainfall conditions, the groundwater table occurs at about 5 to 15 ft-bgs in the vicinity of Koppers Pond. For example, at Well MW-112S, located approximately 250 feet from the northeast corner of the pond (Figure 5), 19 water level measurements collected between December 1996 and July 2006 showed a range of groundwater elevations of 881.0 to 887.4 ft-msl (Cummings/Riter, February 26, 2007). The ground surface elevation at Well MW-112S is 894.1 ft-msl (Westinghouse, December 4, 1997).

Accordingly, the pond recharges groundwater at most times of the year, but may receive groundwater discharges under certain conditions. On a regional scale, however, it does not appear that Koppers Pond is either a significant source of groundwater recharge or a significant discharge location for shallow groundwater.

2.5 LOCAL LAND USE

The pond is surrounded by areas of vacant land and active industrial property. Immediately to the north and northeast is the Old Horseheads Landfill, and to the south is the Kentucky Avenue Well. Manufacturing facilities operated by Hardinge and the Fairway Spring Co. are located to the southeast and east, respectively. Norfolk Southern Corporation (Norfolk Southern) railroad tracks are located to the west. The pond is located on parcels owned by Hardinge, the Village of Horseheads, and the EWB (Figure 3).

The Industrial Drainageway channel is bounded by Norfolk Southern railroad tracks to the west and industrial and commercial properties on the east. These industrial and commercial properties include the Chemung County Department of Public Works (DPW) maintenance facility and the Old Horseheads Landfill.

Public access is not provided to the pond, and no recreational or other use of the pond is authorized by the property owners. Koppers Pond itself is not currently enclosed by fencing, but access to the pond is impeded by intervening railroad tracks and partially fenced industrial and governmental properties. "No Trespassing" signs are posted at the Hardinge property. The presence of sporadic litter with commercial goods (e.g., beverage containers) and off-road vehicle tracks (primarily at the Old Horseheads Landfill) indicate the area is, at times, visited by trespassers. On occasion, persons have been observed bank fishing in Koppers Pond. Both the Village of Horseheads and Hardinge (i.e., the two primary property owners) have recently expressed their intent to increase security measures to discourage trespassing at Koppers Pond.

Several industrial facilities may have, over the years, played an important role in the development of Koppers Pond. These facilities are described in the following sections.

2.5.1 Former Westinghouse Horseheads Plant Site

Westinghouse constructed the Horseheads facility on former farmland and began operations in 1952. This plant developed and manufactured television picture tubes, vacuum switches, and similar electrical products. The plant was expanded several times after its original construction.

In 1985, Westinghouse and the Toshiba Corporation formed an entity (Toshiba-Westinghouse Electric Corporation [TWEC]) to manufacture color television picture screens and related electronic components. After forming TWEC, Westinghouse continued to operate in separate areas in the plant with two divisions. The Imaging and Sensing Technology Division (ISTD) conducted operations in a more central location in the plant, while, while the other Westinghouse division, the Vacuum Tube Interrupter Division, occupied the (eastern) portion of the plant.

Beginning in 1988, Westinghouse sold off its business operations at the Horseheads plant, as follows:

- In 1988, Westinghouse sold ISTD to the Imaging and Sensing Technology Corporation (ISTC), which continued operations (e.g., manufacture of sensor and control products and spectral light sources) at the Site until about 2000.

- In 1989, Westinghouse sold its interest in TWEC to Toshiba Corporation. Toshiba Display Devices, Inc. (TDD), and later MT Picture Display Corporation of America-New York, LLC (MTPDA), continued to occupy a portion of the Horseheads plant for manufacturing operations until 2004.

- In 1994, Westinghouse sold its remaining Horseheads operations (i.e., manufacture of vacuum tube interrupters) to Cutler-Hammer, which continues manufacturing operations in a portion of the plant.

Until April 20, 2007, CBS Corporation (CBS), as the corporate successor to Westinghouse, owned the plant facility and leased space to Cutler-Hammer. On that date, CBS sold the former Westinghouse Horseheads plant site property to Silagi Development and Management, Inc. Cutler-Hammer operations are continuing at this location, and the remainder of the property is slated for use as leased industrial space or redevelopment by the new owner.

2.5.2 Old Horseheads Landfill

The Old Horseheads Landfill forms much of the northern bank of Koppers Pond and the eastern bank of a portion of the lower Industrial Drainageway. Approximately half of the landfill footprint (southern portion) resides in the Town of Horseheads and half (northern

portion) in the Village of Horseheads. The Old Horseheads Landfill was operated from the 1940s until 1973 and reportedly received municipal, commercial, and some industrial solid waste. The landfill was closed for waste disposal in 1975, but no engineered final cover system was constructed at the time of closure.

In the 1980s, USEPA and NYSDEC investigated the Old Horseheads Landfill as a potential source of the TCE that had been found in 1980 in the adjacent Kentucky Avenue Well. The 1990 Supplemental RI (Ebasco Services, Incorporated, July 1990) concluded, however, that this landfill did not contribute TCE to groundwater. In response to an evaluation prepared on behalf of the Town of Horseheads and submitted to NYSDEC in January 1991, the Site was classified as a "Class 3" site on the New York State Registry of Inactive Hazardous Waste Sites, indicating that the Site does not present a significant threat to the public health or environment.

In March 1991, a closure investigation report was submitted to the NYSDEC in support of an application for a Landfill Closure Grant under Title 5 of Article 54 of the Environmental Conservation Law. This application was not approved by New York State.

In 1997, accumulated sediment from the Halderman Hollow Creek storm water detention basin (Figure 2) was removed and the base of the basin was lowered to increase the floodwater detention capacity of the basin. In this effort, approximately 36,000 cubic yards of excavated soils and sediment were removed from the basin and placed as soil cover within the Village of Horseheads portion (i.e., northern area) of the Old Horseheads Landfill. No other remedial activities have been conducted at the Site.

2.5.3 Koppers Company, Inc. Facility

In October 1953, Koppers Company, Inc., now known as Beazer East, Inc. (Beazer), purchased an 87± acre parcel in Chemung County and subsequently constructed its Horseheads Wood Treating Plant (the "KCI-Horseheads Plant") on the property in 1953. Operations began in 1953 or 1954. The KCI-Horseheads Plant ceased operations in 1963 and was dismantled. The property was later acquired by the City of Elmira and Hardinge.

The KCI-Horseheads Plant pressure treated wood. From a review of available records, it appears that the only preservative used in pressure-treating operations at the plant was creosote, and it appears that railroad cross and switch ties were the primary wood products treated.

2.6 ECOLOGICAL SETTING

2.6.1 Open Water

The open water area of Koppers Pond is comprised of a shallow (three- to six-foot deep) warm water lake. The bottom substrate is silty (mucky) and soft over much of the pond. The thickness of the silty sediments is not known, but, based on information collected during past sediment sampling events, is estimated to be on the order of two feet. Debris, such as shopping carts, tires, automobiles, and metal drums, has been observed in the past in and around the pond, but, more recently, the pond area is generally free of this type of debris. Two standing utility poles are located within the open water of the pond and are apparently in use.

2.6.2 Terrestrial Vegetation

The northern and western edges of the pond are vegetated primarily with deciduous trees, and the southern and eastern edges are mostly vegetated with grasses and herbaceous plants. The banks of the Industrial Drainageway are vegetated by occasional cottonwood trees and scrub vegetation.

Dominant tree species in the deciduous woods to the north and west of the pond include cottonwood, willow, sugar maple, and quaking aspen. Shrub species in the deciduous forest include honeysuckle and sumac, and teasel, thistle, and mullein are found in the herbaceous layer.

The open-field cover type on the south and east sides of the pond includes the EWB property around the Kentucky Avenue Well and maintained lawn areas that extend to the Hardinge plant facility. This cover type consists of grasses and forbs in the herbaceous layer, with scattered honeysuckle and brambles in the shrub layer. A scrub-shrub upland community dominated by honeysuckle, brambles, and sumac lies between the two outlet channels.

Two areas (one along the south side and the other at the tip of the western arm) of the open water area are composed of emergent marsh. These are shallow water areas and are largely vegetated with wetland species. The northern area was mapped as an emergent palustrine wetland in the wetland delineation survey conducted as part of the remedial design for the Industrial Drainageway remediation (Hails, July 2001).

2.6.3 Wildlife

Wildlife species reported to inhabit the pond include muskrat, beaver, turtle, green frog, and various fish species (e.g., white sucker, common carp, black crappie, and pumpkinseed). Unidentified minnow-sized fish have been observed in the outlet streams. Terrestrial species that utilize the pond area are believed to include eastern cottontail, woodchuck, raccoon, white-tailed deer, and a variety of birds. Field observations made during past ecological investigations of the Koppers Pond area had noted that amphibians and aquatic insects were scarce or missing from habitats in and around Koppers Pond, although a comparison to nearby ponds of similar size and bathymetry was not conducted. Current conditions with respect to these species are not known.

3.0 SUMMARY OF PREVIOUS INVESTIGATIONS

This section summarizes data and information developed in previous investigations of the Kentucky Avenue Wellfield Superfund Site that defined the nature and extent of COPCs found in environmental media associated with Koppers Pond. Also included in this section are discussions of the fate and transport of these COPCs in the environment. It is recognized that conditions in Koppers Pond are dynamic, and many aspects of the pond have likely changed since the time data were collected and evaluated in previous investigations. Nonetheless, the information provided in this section provides focus and perspective in planning the requirements for RI data-gathering activities.

3.1 SOURCES

This section discusses the known and potential sources of the COPCs previously found in the surface water, sediments, and fish in Koppers Pond. This information has been compiled from the findings of the Operable Unit 3 RI, evaluations associated with the remedial design and remedial action for the Industrial Drainageway, and other recent studies.

3.1.1 Historical Sources

3.1.1.1 Former Westinghouse Horseheads Plant

Discharges from the former Westinghouse Horseheads plant site were historically a source of several of the COPCs now observed in Koppers Pond. As indicated in Section 2.5.1, the history of operations at that plant site is somewhat complicated, and the history of waste water treatment operations, treated discharges, and discharge permitting is more complex. The following paragraphs briefly summarize the waste water discharge history of the former Westinghouse Horseheads plant site, and Table 1 provides a summary chronology. The Operable Unit 3 RI provides an inventory (through early 1996) of the various discharge permits and the results of effluent monitoring associated with those permits.

With the initial construction of facilities in 1952, Westinghouse installed a waste water treatment plant to control the pH of waste waters, but there were no provisions at that time to collect the solids generated by this treatment. The plant waste water treatment

system was upgraded in the late 1950s to provide lime addition with separation of the precipitated calcium fluoride. Westinghouse again upgraded its waste water treatment facilities in 1967 to improve metals precipitation and clarification processes. Until 1994, when it completed the sale of all manufacturing operations at the Horseheads plant, Westinghouse operations discharged treated waste waters via designated Outfall 001W (Figure 6).

When Westinghouse operations began in 1952, waste water discharges were not regulated; however, in 1957, Westinghouse submitted a permit application to the New York State Department of Health (NYSDOH) to operate the waste water treatment facilities at the Site. With passage of the Federal Water Pollution Control Act Amendments of 1972 (FWPCA), the National Pollutant Discharge Elimination System (NPDES) permitting process was initiated. Under the NPDES program, effluent limitations were established for specific types of waste water discharges. NYSDEC was granted primacy for permitting and began issuing State Pollution Discharge Elimination System (SPDES) permits under FWPCA authority. Westinghouse applied for an SPDES permit, which was received in March 1973. This permit placed effluent limitations on pH, suspended solids, fluoride, chromium, copper, lead, and zinc. Subsequent permits renewed through 1996 placed effluent limitations on heavy metals (e.g., cadmium, chromium, copper, lead, nickel, silver, and zinc), cyanide, fluoride, TCE, and other parameters.

In August 1987, TWEC was issued a separate permit for its process and cooling waste water discharges through an outfall designated 001T (Figure 6). This permit was subsequently revised in 1990 by TDD and renewed at varying times until treated waste water discharges were terminated in 2004. TDD, and later MTPDA, operated its own waste water treatment system at the Horseheads plant site. Effluent limits for the treated waste water from this system included those for aluminum, arsenic, chromium, copper, cyanide, fluoride, iron, lead, nickel, silver, zinc, and TCE.

From 1988 through 1996, waste water discharges from ISTC were conveyed both to the Westinghouse waste water treatment plant and to the TWEC/TDD waste water treatment plant and discharged at Outfalls 001W and 001T, respectively.

From 1994 through 1996, Cutler-Hammer also conveyed waste waters to the Westinghouse on-Site waste water treatment plant that discharged to Outfall 001W. In 1996, the following changes were made (Figure 6):

- Westinghouse closed its on-Site waste water treatment plant;
- Cutler-Hammer and ISTC plating waste waters were rerouted to the Chemung County Sewer Authority;
- Westinghouse directed the discharges from the newly installed barrier well treatment facility installed under Operable Unit 2 (i.e., water not needed in plant site manufacturing operations) to a newly designated Outfall 001W;
- Westinghouse separated its discharge lines from those associated with Cutler-Hammer operations and redirected boiler blowdown, non-contact (compressor) cooling water, and deionized water tank backwash discharges to designated Outfall 002W;
- Cutler-Hammer discharges (e.g., process and cooling waters) continued through "old Outfall 001W" under an SPDES permit issued to Cutler Hammer (for clarity, the "old Outfall 001W" is referred to as "Outfall 001CH"); and
- ISTC process and cooling water discharges were routed to Outfall 001CH, although some ISTC waste waters were also discharged discharge via Outfall 001T.

The Cutler-Hammer SPDES permit (Outfall 001CH), which is still active, provides effluent limitations on several metals, including copper, lead, nickel, and zinc.

Following MTPDA's cessation of operations at the former Westinghouse Horseheads plant site, CBS terminated the discharges of non-contact (compressor) cooling water, and deionized water tank wash from Outfall 002W, and, with the reduced demand for water from manufacturing operations, the barrier well treatment facility effluent has been primarily discharged directly to Outfall 001W. Cutler-Hammer continues to use a small amount of the barrier well discharge water; usage rates vary from approximately 1.8 million gallons per month (60,000 gallons per day) in the winter to 7.8 million gallons

per month (160,000 gallons per day) in the summer. The barrier well effluent contains very low to non-detectable levels of metals and other monitored constituents. Outfall 002W now only receives storm water runoff from certain plant site roofs.

Beginning in early 1995, a whitish-brown floc was observed in the flow of the Industrial Drainageway, and this floc was carried to Koppers Pond. Subsequent investigations by NYSDEC and USEPA found that the floc entered the Industrial Drainageway at the Chemung Street Outfall. NYSDEC described the floc as a microbial material that formed under particular conditions of temperature and dissolved oxygen and the presence of phosphate compounds. Chemical analyses (NYSDOH, May 1997) showed the presence of lead and other metals (e.g., copper and zinc). The floc apparently served as a substrate for accumulating metals, and the metals associated with the floc were likely absorbed from metals already present in the permitted discharges. NYSDEC further postulated that the floc material had accreted on the inside of the underground piping that connected the TDD discharge to the Chemung Street Outfall.

TDD modified its waste water treatment system in an effort to curtail the floc formation. These efforts, which were completed in 2001, appeared to reduce the quantity of floc observable in the Industrial Drainageway, but did not entirely end its occurrence. Analytical testing conducted at various times suggests that the lead and other metals levels originally associated with the floc did not persist.

3.1.1.2 Old Horseheads Landfill

The extent to which the Old Horseheads Landfill may have contributed COPCs to Koppers Pond is not known. It is apparent from review of historical topographic maps and aerial photographs that landfill operations filled the northern portion of the open-water area of the pond that had formed in the early 1950s, and there is the potential for the southern portion of the landfill to be in hydraulic communication with the pond. Surface water runoff from the landfill could carry COPCs for subsequent deposition in Koppers Pond. Investigations conducted for the preparation of the 1991 closure investigation report (Fagan Engineers, March 1991) indicted that only construction and demolition debris was placed as fill south of the overhead electric lines near the Village of Horseheads boundary and that no municipal or industrial waste was placed south of the electric lines in the lower lying wet areas (Figure 5).

The Supplemental RI completed in 1990 (Ebasco Services, Incorporated, July 1990) included some soil sampling at the Old Horseheads Landfill. Pesticides, polychlorinated biphenyls (PCBs), and polynuclear aromatic hydrocarbons (PAHs) were all detected in at least one soil sample. PCB concentrations ranged to 300,000 micrograms per kilogram (µg/kg) (300 milligrams per kilogram [mg/kg]) in a subsurface soil sample (i.e., SB-9A, 10 to 12 ft-bgs); surficial concentrations were much lower. The PCBs detected at SB-9A were identified as Aroclor 1248. The pesticides β-BHC (benzene hexachloride, also known as hexachlorocyclohexane or lindane), heptachlor, Endosulfan I, Endosulfan II, and endosulfan sulfate were detected, with concentrations of individual compounds ranging to 3,800 µg/kg (i.e., Endosulfan I at SB-8A, 15 to 17 ft-bgs). At one boring, the total PAHs concentration in a subsurface sample was in excess of 360,100 µg/kg (i.e., SB-6A, 5 to 7 ft-bgs). Borings SB-6A, SB-8A, and SB-9A were all located north of the overhead electric lines near the Village of Horseheads boundary and north of the area shown in the March 1991 report for construction and demolition debris disposal (Figure 5). Historical groundwater monitoring data did not show elevated COPC levels in Well MW-103S (fka Well MW-12S) near the southern end of the landfill (i.e., the well closest to the pond).

3.1.1.3 KCI-Horseheads Plant

The extent to which the former KCI-Horseheads Plant may have contributed COPCs to Koppers Pond is not known. Based on a review of historical maps, aerial photographs, plant plans, and other information, the plant appears to have been situated to the south and southeast of the present-day Koppers Pond (i.e., on the downstream side of the pond). Former wood-treating operations appear to have occurred in the area immediately north of the terminus of Kentucky, Michigan, and Vermont Avenues.

According to historical records, excess creosote from the plant's treatment process was recycled for reuse, aqueous wastes were evaporated in plant process tanks, non-recyclable creosote wastes were burned in the plant boiler, and the plant did not dispose of wastes on Site (Ecology & Environment, Inc., December 1991). The KCI-Horseheads Plant was reportedly served by public water, and sanitary wastes from the plant may have been treated through an on-Site septic system, as historical plant design plans note a "drain field" connected to the plant's "office" building near the southern end of the plant property.

None of the historical maps, plans, or documents reviewed in Beazer's files references "Koppers Pond." According to a property survey map dated August 7, 1953, there was an area referred to as a "swamp" that lay to the north and northwestern portions of the KCI-Horseheads Plant property. Plant maps and plans do not reference any operational structures situated in these northern and northwestern portions of the plant property; instead, available maps and plans place all plant buildings, structures, and features in the southern and southeastern portions of the plant property. The only potential plant discharge point located during a review of historical files was one line marked on a 1954 plant plan as a "new ditch" that terminated at the southern tip of the plant property near the confluence of "Rockwell's Creek" and "Hartman Hollow Creek." This 1954 plant plan does not reference or explain the purpose or use of such "new ditch" by the KCI-Horseheads Plant.

In 1989 and 1991, respectively, NYSDEC completed Phase I and Phase II assessments of the KCI-Horseheads plant site and concluded that there was no evidence of past on-Site hazardous waste disposal. In 1992, the Site was deleted from the NYSDEC registry of known or suspected hazardous waste disposal sites.

3.1.2 Ongoing Sources

Treated process discharges from the Cutler-Hammer operations at the former Westinghouse Horseheads plant site (i.e., Outfall 001CH) remain a potential source of ongoing contributions of COPCs to Koppers Pond. Runoff from local industrial and commercial facilities and local roadways may also contain COPCs, but such contributions have not been quantified. Leachate from the Old Horseheads Landfill has the potential for affecting Koppers Pond on an ongoing basis, but this contribution also has not been quantified.

Floc material continues to be observed in the Industrial Drainageway, but in quantities well below what had previously been observed. It is not known whether this floc is continuing to form or whether it is flaking off from material formed in the past on the interior of the underground piping connecting Outfall 001T at the former Westinghouse Horseheads plant site to the Chemung Street Outfall. Although there is no known comprehensive database, the sporadic sampling data that have been developed suggest that this floc no longer exhibits the levels of lead and other metals previously ascribed to

it. This observation is consistent with the understanding that metals loadings have significantly decreased from discharges from the former Westinghouse Horseheads plant site and the hypothesis that the floc may serve as a substrate that absorbs dissolved metals already present in the water.

3.2 SURFACE WATER

3.2.1 Surface Water Sampling Data

In 1994 and 1995, the Operable Unit 3 RI included two rounds of surface water sampling from the Industrial Drainageway and Koppers Pond to characterize water quality and to identify sources of COPCs. Three of the sampling locations (i.e., Nos. 15, 17, and 18) were in Koppers Pond, and three were located in the outlet channels (i.e., Nos. 16, 19, and 20). Sample No. 21 (1995 sampling only) was located in the Industrial Drainageway near its discharge to Koppers Pond. Table 2 lists these seven sampling locations and the water characterization data collected at each at the time of sampling. Figure 3 shows these sampling locations.

The first round of surface water samples collected for the Operable Unit 3 RI (i.e., those from June 1994) were analyzed for target compound list (TCL) volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), pesticides and PCBs, target analyte list (TAL) metals, and total cyanides. Selected samples were also analyzed for fluoride. Surface water samples from the second event (i.e., June 1995) were analyzed for TAL metals, total suspended solids, and hardness. Table 3 provides a summary of the analytical data from the Koppers Pond surface water sampling.

In addition to the Operable Unit 3 RI data, CDM Federal Programs Corporation (CDM) collected surface water quality information in 1998 during the sediment sampling conducted in support of its ecological risk evaluation of Koppers Pond (CDM, February 1999). These 1998 CDM surface water characterization data are included in Table 2. Similarly, during fish sampling conducted in July 2003, Civil & Environmental Consultants, Inc. (CEC) collected water quality characterization data at four locations in Koppers Pond. These data are also included in Table 2.

3.2.2 Surface Water Data Assessment

All of the surface water characterization and quality data provided in Tables 2 and 3 were collected at a time when treated industrial waste waters were being discharged from the former Westinghouse Horseheads plant site at rates higher than present discharge rates. With discharges now having been reduced, and the reported data may not be representative of current conditions. Surface water sampling will be performed as described in this Work Plan, and the surface water data used in the Site evaluations will be representative of the pond in its current condition. Some general observations of the available historical information are nonetheless useful.

The Operable Unit 3 RI data showed depressed dissolved oxygen readings in samples from the lower Industrial Drainageway, Koppers Pond, and the outlet channel (Table 2). These data also showed a wide variation in pH, with values ranging from 4.0 at Sample Location 18 in the pond to 8.9 in the outlet channels (Sample Locations 19 and 20). Such water quality conditions represent potential stressors to the aquatic ecosystem. Subsequent sampling by CDM similarly showed somewhat depressed dissolved oxygen levels in the western portion of the pond near the discharge of the Industrial Drainageway, but higher levels throughout the remainder of the pond and discharge channels. CDM's pH readings all showed slightly alkaline water, with values ranging from 7.63 to 8.57 and a geometric mean of 8.03. The 2003 CEC data were generally consistent with the CDM 1999 data, although CEC did not identify an area of depressed dissolved oxygen in the western portion of the pond. The variation in dissolved oxygen among these three sampling events may have reflected associated changes in the discharges to the Industrial Drainageway; however, differences in water temperature, sampling personnel, and other factors make such conclusions tentative.

As shown in Table 3, TCE, which was a regulated constituent in the SPDES permit from an outfall at the former Westinghouse Horseheads plant site, was found in low (estimated) concentrations in some samples, but TCL VOCs were not present at elevated concentrations in any of the surface water samples associated with Koppers Pond. This result would be expected as VOCs, because of their volatility and typically low solubility, are usually not important constituents in shallow surface water systems. Fate processes

normally result in these compounds being volatilized from flowing water systems, especially shallow streams with riffle sections, such as the Industrial Drainageway (Lyman, et al., 1982).

No TCL SVOCs or PCBs were detected in the surface water samples associated with Koppers Pond. Such compounds typically exhibit low solubility and are hydrophobic. Their absence from surface water would be expected.

The pesticide compounds α-BHC and β-BHC were detected in some surface water samples; however, in all cases, the data were qualified due to lack of reproducibility in dual gas chromatography/mass spectrometry analyses. These compounds are associated with the pesticide lindane, which prior to use restrictions effective in 1983, was widely applied as an insecticide for mosquitoes and parasites (Sittig, 1981; Agency for Toxic Substances and Disease Registry, 1997). Their detection in surface water at Koppers Pond was likely attributed to historical area-wide or local applications.

The surface water quality in the samples associated with Koppers Pond reflected the influence of the permitted treated waste water discharges to the Industrial Drainageway. Surface water samples showed the presence of several metals that are regulated constituents in past SPDES permits (e.g., aluminum, chromium, lead, zinc) as well as fluoride. Cadmium and copper were not detected in samples from Koppers Pond or its outlet channels, but were found in the Industrial Drainageway sample near its discharge into Koppers Pond (Sample Location 21). Because of the changes with respect to the permitted discharges that were occurring at the time of sampling versus those which are currently active, comparisons of constituents in prior sampling to ambient water quality criteria under present conditions are not germane, and current data are required for meaningful comparisons.

3.3 SEDIMENTS

3.3.1 Operable Unit 3 Remedial Investigation Data

The Operable Unit 3 RI also included two rounds of sediment samples collected at the same time as the corresponding surface water samples. The initial round of samples was collected in 1994 and included six locations in Koppers Pond and its outlet channels

(Figure 3). These sediment samples were collected to a maximum depth of 24 inches and were composited throughout the depth of recovery. Collected samples were analyzed for TCL VOCs, SVOCs, pesticides and PCBs, and for TAL metals and total cyanides.

The second round of Operable Unit 3 RI sediment sampling was conducted in May and June 1995 to further characterize Site conditions. These second-round sediment samples were originally to be analyzed for TCL SVOCs, PCBs and pesticides, mercury, and total organic carbon (TOC). Analyses for metals were not planned because such metals were listed as permitted discharge parameters on the two SPDES permits for the former Westinghouse Horseheads plant site and had already been detected in the earlier sediment samples. Prior to commencement of the June 1995 sampling activities, however, the whitish-brown floc was observed floating in the Industrial Drainageway (Section 3.1.1.1). This material was first reported to NYSDEC in March 1995 and continued to be observed in the Industrial Drainageway throughout the remainder of 1995 and 1996. Analysis of this material showed it to contain several metals, and, based on these analytical results, USEPA requested that the second-round sediment samples also be analyzed for cadmium, chromium, and lead. Also, unlike the earlier samples that were composites collected over depths ranging to 24 inches, the second-round sediment samples were collected from the uppermost 6 inches of encountered material.

Tables 4 through 6 provide the results of the Operable Unit 3 RI sediment sampling in Koppers Pond. Table 4 presents results on TAL metals and cyanides. Table 5 provides the data for TCL VOCs and SVOCs, and Table 6 presents the data for TCL pesticides and PCBs.

3.3.2 1998 CDM Sampling Data

In support of its draft baseline ecological risk assessment (BERA), CDM collected sediment samples from 14 locations in Koppers Pond and adjacent waterways in August 1998. The sediment samples were typically collected from the uppermost six inches of the sediment surface, although vertical sediment profiles with multiple samples were collected at two locations. Collected sediment samples were analyzed for TAL metals and cyanide and for TCL pesticides and PCBs. These data are included in Tables 4 and 6, respectively. Table 6 also includes the results of TOC analyses conducted on these samples.

In addition to direct chemical analysis, CDM conducted toxicity testing of sediments using the *Hyalella azteca* (amphipod) 10-day survival test and the *Chironomus tentans* (midge) 10-day survival and growth test, in accordance with USEPA (June 1994) methods (i.e., Method 100.1 and 100.2, respectively) used at that time. This testing was designed to assess the potential toxicity of pond sediments to benthic macroinvertebrates. CDM also performed a benthic macroinvertebrate community survey and analysis to assess potential toxicity of the pond sediment to these organisms.

3.3.3 Sediment Data Assessment

The sediment data associated with Koppers Pond from the Operable Unit 3 RI and the supplemental 1998 CDM data showed the presence of metals, several of which exhibited concentrations above NYSDEC (January 1999) screening levels. Metals concentrations were generally higher in the western portion of the pond near the discharge from the Industrial Drainageway and lower in the outlet channels of the pond. This pattern is consistent with the hydraulics of these surface water bodies whereby Koppers Pond has acted as a sediment trap and settling basin for suspended and precipitating dissolved metals present in the surface waters of the Industrial Drainageway.

The samples from Koppers Pond and its outlet channels did not show significant detections of VOCs, and no TCE was detected in any of these samples. Detected VOCs were carbon disulfide, a naturally occurring compound found in anoxic environments, methylene chloride, and toluene. All detections were estimated values at concentrations below the corresponding analytical quantitation limits.

SVOCs detected in Koppers Pond sediments were primarily PAHs and, to a lesser degree, phthalates. The phthalates found in the sediment samples are found in a wide variety of plastic products and could be the result of contamination introduced in sampling or laboratory analysis.

The PAHs detected in the sediment samples are found in asphalt, motor vehicle fuels, lubricants, and other related coal and petroleum-based materials, including coal-fired boiler bottom ash ("cinders") (Verschueren, 1983; Sittig, 1981). Their presence in the pond may be related to surface water runoff from nearby commercial and urban areas, including the Old Horseheads Landfill and the cinder storage areas at the Chemung

County DPW maintenance facility. The highest observed concentration in the western portion of Koppers Pond was found in Operable Unit 3 RI Sample 20B at 3.8 mg/kg total PAHs, and, in the northeast corner of the pond, total PAHs at Operable Unit 3 RI Sample Location 17 were 3.2 mg/kg total PAHs. PAHs may also be related to the former KCI-Horseheads plant in the area to the east and south of the pond. Information is lacking regarding the operation of the former KCI-Horseheads plant, however, and no definitive conclusions regarding the contribution of that plant to the observed PAHs can be drawn.

A variety of pesticides were found in various sediment samples associated with Koppers Pond, and several of these pesticides were present in at least one sample above NYSDEC (January 1999) sediment screening values based on benthic aquatic life chronic toxicity or wildlife bioaccumulation. Detections of pesticides were somewhat sporadic (i.e., not pervasive across all samples), and most of the values were detected below quantitation limits. Endrin, 4,4'-DDT (dichlorodiphenyltrichloroethane), and γ-chlordane were detected most frequently. Like the α- and β-BHC found in surface water samples, the detected pesticides are all insecticides that were typically applied over large areas of cropland and open water. Their presence most likely represented residuals from historical area-wide or local applications. The use of most of these detected pesticides (e.g., chlordane, DDT, endrin, lindane) has either been severely restricted or banned entirely for many years dating back to the 1970s and early 1980s.

PCB Aroclor 1254 was detected in approximately two-thirds of the sediment samples collected from Koppers Pond and its outlet channels, with detected concentrations ranging from 110 to 4,500 µg/kg. A duplicate of the sample showing the highest concentration (i.e., CDM Sample Location 20) exhibited 1,100 µg/kg (i.e., CDM Sample Location 11). The NYSDEC (January 1999) sediment screening value for PCB Aroclor 1254 is 1.4 micrograms per gram TOC. Based on the observed TOC concentrations, NYSDEC sediment screening levels for Koppers Pond would range from 81 to 288 µg/kg.

The source of the PCBs in Koppers Pond is not well understood. The operations at the former Westinghouse Horseheads plant did not involve the manufacture or assembly of fluid-filled electrical equipment, and, in the extensive investigations of potential source areas at the former Westinghouse Horseheads plant site conducted under Operable

Unit 3, the highest PCB concentration found in any on-Site soil or waste sample was 2.4 mg/kg of Aroclor 1260. Similarly, the highest concentration of Aroclor 1254 found in any such on-Site soil or waste sample was 0.79 mg/kg.

Prior to the remediation of the Industrial Drainageway completed in early 2003, PCBs had been found in the Industrial Drainageway sediments; the maximum PCB concentrations in the removed materials exceeded 50 mg/kg (Cummings/Riter Consultants, Inc., April 2001). The presence of PCBs in Koppers Pond sediments may be, at least in part, the result of the transport of these compounds as suspended or bed-load sediments from the drainageway. In addition, PCBs apparently were disposed of in the Old Horseheads Landfill, and these disposal activities may have impacted Koppers Pond.

Despite the chemical data for sediments showing inorganic and organic constituent concentrations above screening levels based on aquatic organism toxicity, the results of the Site-specific toxicity testing showed little impact. This testing, conducted as part of the draft BERA, specifically was designed to assess the potential toxicity of pond sediments to benthic macroinvertebrates. In the 10-day survival test, *Chironomus tentans* showed no significant reduction in survival as compared to the control group for any of the 14 pond samples tested and growth in all pond sediment samples was greater than in laboratory controls. Only one of the 14 sample locations (i.e., Sample Location 13 at the mouth of the Industrial Drainageway where it enters Koppers Pond) showed toxicity to *Hyalella azteca*. This sampling location is just south of the extent of sediments removed as part of the completed (Operable Unit 3) Industrial Drainageway remediation (Figure 3).

The associated benthic macroinvertebrate survey and analysis designed to assess potential chronic toxicity of the pond sediment to these organisms showed moderate to severe sediment impacts using the metrics provided by USEPA (May 1989) guidance. Several of the applied metrics, however, are applicable to flowing streams, whereas Koppers Pond is a shallow, eutrophic pond with very low horizontal flow velocity (i.e., less than 0.01 feet per second). Because of these differences, and without a reference pond for comparison, definitive conclusions cannot be drawn regarding sediment toxicity to benthic macroinvertebrates.

3.4 FISH

3.4.1 Sampling Data

In sampling conducted in 1988, NYSDEC reported the detection of PCBs in largemouth bass and carp collected from Koppers Pond. These findings led to the issuance of a fish advisory for the Pond by NYSDOH; the NYSDOH advisory, which is still in effect, is for carp with a recommendation to eat no more than one meal per month. To follow-up these earlier data, supplemental fish sampling was identified as a task for the Operable Unit 3 RI.

After an initial (spring 1994) attempt to collect fish samples using normal fishing techniques was unsuccessful, fish sampling from Koppers Pond using electroshocking techniques was completed in June 1995 as part of the Operable Unit 3 RI. This sampling resulted in the collection and tissue analysis from 15 fish samples (i.e., 6 white sucker and 9 common carp). Of these, three of the carp samples (i.e., CC-07, CC-08, and CC-09) were composited into one sample for analysis because of the limited sample size available from these discrete fish tissue samples. Skinless fish fillets from collected specimens were analyzed for TCL VOCs, SVOCs, pesticides, and PCBs and TAL metals and cyanide, although limited sample size did not allow for analysis of all parameters in all samples. The developed analytical data are presented in Table 7.

In 2003, CEC, under contract to Viacom Inc. (at that time the corporate successor to Westinghouse) conducted fish sampling in Koppers Pond to provide updated information on PCB and metals concentrations in fish tissue. Fish were collected using electroshocking techniques, resulting in a total of 24 fish samples for analysis. Collected species included both bottom-feeding (i.e., common carp and white sucker) and pelagic species (i.e., largemouth bass, pumpkinseed, black crappie, and green sunfish). The samples of fish for potential human consumption (i.e., common carp, white sucker, and largemouth bass) were prepared as skin-on fillets with the belly flap included, in accordance with NYSDEC (October 2002) procedures. Smaller fish species for ecological evaluation (i.e., pumpkinseed, black crappie, and green sunfish) were analyzed as whole-body samples. Table 8 presents the results of these analyses.

3.4.2 Fish Data Assessment

The 1995 fish sampling data showed that bottom-feeding species in Koppers Pond did not contain elevated concentrations of TCL VOCs or SVOCs, including the PAHs identified in some sediment samples. PCB Aroclor 1254 was detected in 10 of 12 analyzed samples, with detected concentrations ranging from 93.1 to 537 µg/kg. The 2003 U.S. Food and Drug Administration (FDA) advisory level for total PCBs in fish is 2,000 µg/kg (21 Code of Federal Regulations 109.30). The FDA level is designed to protect consumers of market fish and not anglers who may have a higher fish consumption level.

The results of the 2003 fish sampling showed higher levels of PCBs than were detected in the 1995 sampling. PCBs were detected in all fish samples, with concentrations ranging from 270 to 2,400 µg/kg. Differences between the analytical findings from the 1995 samples (skinless fillets) and the 2003 samples (skin-on fillets and whole body samples) are believed to result primarily from the dissimilarities in sample preparation techniques used in these two sampling events, and are not believed to indicate that PCB levels are increasing in the pond ecosystem.

3.5 HUMAN HEALTH EVALUATION

Using the data developed under the Operable Unit 3 RI, CDM conducted a baseline human health risk assessment (BHHRA) on behalf of USEPA, publishing its findings in a report dated November 1995. The BHHRA evaluated potential exposure pathways for area residents potentially contacting COPCs in surface waters and sediments in the Industrial Drainageway and Koppers Pond and potentially consuming fish taken from these water bodies.

The BHHRA followed standard protocols as specified in USEPA *Risk Assessment Guidance for Superfund* (RAGS) (December 1989) and examined the consequences of exposure to various COPCs by current and potential future human receptors under a variety of exposure scenarios. Consistent with USEPA guidance, the risk assessment did not address the probability of such occurrences and, as a baseline assessment, did not consider institutional controls or other impediments to the assumed exposure scenarios (e.g., NYSDOH fish advisory for consumption of carp taken from Koppers Pond) in calculating potential exposure to the reasonably maximum exposed individual.

The risk assessment used combined data sets from both the Industrial Drainageway and Koppers Pond for the evaluation of potential risks associated with exposure to surface water and sediments. This risk assessment examined potential carcinogenic and non-carcinogen impacts associated with ingestion of and dermal contact with surface water and sediments during primary recreational activity (e.g., wading or swimming) at a frequency of 24 days per year. The BHHRA showed potential carcinogenic risks associated with individual exposure pathways ranging from 2.8×10^{-7} for surface water exposure pathways to 1.5×10^{-6} for contact with sediment. The sediment contact risk was driven by materials in the Industrial Drainageway; Koppers Pond sediments did not contribute to this potential risk. The estimated hazard indices (HIs) for non-carcinogenic exposures via all exposure pathways fell well below USEPA's target of 1.0. On this basis, USEPA concluded that direct exposure to surface waters and sediments associated with Koppers Pond did not pose an unacceptable human health risk.

The human health risk assessment also examined potential risks associated with consumption of fish taken from Koppers Pond. For this evaluation, the risk assessment used the fish tissue data gathered in the 1995 Operable Unit 3 RI sampling (see Section 3.4, Table 7) and used a fish consumption rate of 0.5 pound of fish per meal for 50 meals per year. Based on these assumptions, the human health risk assessment estimated a potential incremental excess lifetime cancer risk of 3.8×10^{-4} associated with fish consumption. The estimated potential HI for non-carcinogenic risk was 6.9. The potential carcinogenic risk associated with fish consumption was due to the presence of PCBs and arsenic in fish tissue samples. The potential non-carcinogenic HI of 6.9 was almost entirely the result of PCBs (Aroclor 1254). These potential risk levels are above USEPA's target risk range and drove the requirements for remediation of Industrial Drainageway sediments, where PCBs levels were higher, under Operable Unit 3.

Based on the results of the fish tissue sampling conducted in 2003, CEC recalculated the potential human health risk specific to the consumption of fish taken from Koppers Pond. In this evaluation, CEC used the exposure scenarios and assumptions that were presented in the BHHRA (CDM, November 1995), but considered two different fish consumption rates:

- Central Tendency Case: 0.005 kilogram per day (kg/day), corresponding to the mean value for recreational freshwater anglers in New York (USEPA, August 1997); and

- Reasonable Maximum Case: 0.03 kg/day to correspond to the consumption rate used in the CDM (November 1995) BHHRA.

For these two cases, the potential incremental excess lifetime cancer risk ranged from 5.4×10^{-5} for the central tendency case to 1.1×10^{-3} for the reasonable maximum exposure case. Corresponding potential HI values were 2.7 and 21, respectively. The CEC calculations were again "baseline" assessments prepared in accordance with USEPA guidance, including the April 1991 guidance *Role of the Baseline Risk Assessment in Superfund Remedy Selection Decisions* (OSWER Directive 9355.0-30). In the CEC evaluation, neither the central tendency nor the reasonable maximum case considered the NYSDOH fish advisory for consumption of carp taken from Koppers Pond, access limitations, or other institutional or physical controls that indicate actual consumption rates are likely to be substantially smaller than the rates assumed. The pond is relatively small with limited fish populations, and there no evidence of subsistence-level fishing.

3.6 ECOLOGICAL EVALUATION

3.6.1 Screening Level Ecological Risk Assessment

In March 1996, USEPA conducted a screening-level ecological risk assessment (SLERA) that focused on the Industrial Drainageway and Koppers Pond. The SLERA was prepared in accordance with USEPA guidance used at that time (USEPA, September 1994) to assess whether COPCs in the sediments and surface water at the Industrial Drainageway and Koppers Pond area had the potential to adversely impact ecological receptors at the Site. In the SLERA, USEPA screened constituent concentrations developed under the Operable Unit 3 RI against ecological benchmarks and state of New York fish criteria. Following this screening, constituents identified as primary contributors to ecological hazard were then used to characterize potential ecological risk to select receptor species (i.e., blue heron and raccoon).

The results of the SLERA showed that Site-related COPCs were present in sediment and surface water samples collected from the Industrial Drainageway and Koppers Pond at concentrations that have the potential to pose adverse ecological effects. Based on this finding, USEPA then proceeded to conduct a more expansive draft BERA.

3.6.2 Draft Baseline Ecological Risk Assessment

In 1999, under contract to USEPA, CDM completed a draft BERA focused on Koppers Pond. In this study, CDM calculated ecological hazard quotients (HQs) as the ratios of observed concentrations of COPCs in sediment to corresponding sediment screening values and modeled uptake of COPCs from sediment into higher trophic level species (i.e., mink, raccoon, and great blue heron). The draft BERA used the chemical data (i.e., metals, PCBs, and pesticides) collected in the 1998 sampling to update the information available from the Operable Unit 3 RI.

The draft BERA showed that the calculated HQs were greater than 1.0 for several COPCs at many sampling points within Koppers Pond and its outlet channels, and the draft BERA's modeling of potential risks to higher trophic levels showed unacceptable risk to all three target species. Such potential risks were expressed as HIs and calculated based on No Observed Adverse Effects Levels (NOAELs). The primary COPCs contributing to the HIs were metals, including cadmium, chromium, copper, lead, and zinc; the relative portions of the total NOAEL HI contributed by these metals are summarized as follows.

COPC	Great Blue Heron	Raccoon	Mink
Cadmium	1.2%	32.2%	10.2%
Chromium	88.1%	1.4%	10.1%
Copper	0.1%	32.0%	0.6%
Lead	1.7%	29.7%	66.4%
Zinc	5.8%	2.7%	1.7%
Total	96.9%	98.0%	88.9%

HQs associated with PCBs were 0.33 and 0.28 for the great blue heron and raccoon, respectively. For the mink, a mammal that has been found especially sensitive to PCBs, the estimated HQ for PCBs was 71.9, which represented 4.6 percent of the total risk (i.e., calculated NOAEL HI) for the mink.

The 1999 draft BERA modeled uptake and bioaccumulation of metals and PCBs from sediments into fish (instead of using measured fish data) when evaluating potential risk to higher trophic levels. As part of its 2003 assessment, CEC (July 2003) recalculated these potential risks using the actual fish tissue data collected in the 2003 sampling in lieu of the modeled values applied in the draft BERA. Using the actual rather than modeled metals and PCB concentrations in fish resulted in a reduction of the estimated potential HIs for the target species (i.e., great blue heron, raccoon, and mink) by nearly an order of magnitude. For example, the calculated HI based on NOAEL for the great blue heron was reduced from approximately 1,900 to 210 by using CEC's actual metals and PCB concentrations determined in fish tissue sampling rather than the modeled metals and PCB metals concentration from the draft BERA. Using actual fish tissue concentration, PCBs were found to contribute less than 0.04 percent of the risk to the great blue heron, calculated as the NOAEL HI.

4.0 PRELIMINARY CONCEPTUAL SITE MODEL

This section summarizes the PCSM for Koppers Pond based on the review of available and historical information and data (Sections 2.0 and 3.0). The Site model presented in this Section 4.0 is updated from that provided in the PCSM report submitted to USEPA by the Group on February 19, 2007, pursuant to Paragraph 27a(1) of the Settlement Agreement. Revisions are based on comments received from USEPA on that submittal. As the RI proceeds and additional Site data are collected and evaluated, this understanding of Site conditions, COPCs, and the potential risk associated with the presence of these COPCs, are likely to evolve and could change significantly.

In accordance with the Settlement Agreement and pertinent USEPA guidance (March 1987; October 1988), the PCSM is a component of the Work Plan development process and presents information on COPCs, known and suspected sources of these COPCs, migration pathways, human and ecological receptors, and routes of potential exposure. Although several of the same topics are addressed, this PCSM was not prepared to fully vet the issues to be addressed in developing the conceptual site model as part of problem formulation under the USEPA (June 1997) *Ecological Risk Assessment Guidance for Superfund* (ERAGS). The problem formulation step for ecological risk evaluations will be addressed as evaluations are conducted under ERAGS Steps 1 and 2 in developing the revised SLERA (Section 6.6.2).

4.1 SOURCES

4.1.1 Historical Sources

Historically, the Industrial Drainageway was fed by discharges from the former Westinghouse Horseheads plant site, and these discharges, which occurred over more than 40 years, are one source of the metals found in the Koppers Pond sediments. A number of the metals identified in the various discharge permits for the former Westinghouse Horseheads plant site operations are also observed in sediments. Other sources of metals include industrial and urban runoff, as described in Section 4.1.2 below.

The source of the PCBs in Koppers Pond is not well understood. It appears that the PCBs entered Koppers Pond via the Industrial Drainageway in the suspended or bed-load sediments transported by the drainageway.

The pesticides found in the surface water and sediment of Koppers Pond have no known point source. These pesticides, which are primarily insecticides, may have been associated with mosquito control or other area-wide or local applications. Many of the pesticides that are COPCs have been significantly restricted in their uses or banned entirely, and ongoing sources of these constituents to the pond would not be expected.

PAHs are considered of secondary concern in Koppers Pond sediments because of lower concentrations, less pervasive occurrence, and the absence of PAHs in fish tissue samples analyzed as part of the Operable Unit 3 RI. The PAHs detected in the sediment samples are found in asphalt, motor vehicle fuels, lubricants, cinders, and other related coal and petroleum-based materials. Their presence in the pond may be related to surface water runoff from nearby industrial, commercial, and urban areas, including the Old Horseheads Landfill and, possibly, operations at the former KCI-Horseheads plant.

4.1.2 Ongoing Sources

At present, discharges to the Industrial Drainageway from the former Westinghouse Horseheads plant site are those from the Cutler-Hammer outfall, the treated effluent from the barrier well groundwater treatment facility, and storm water runoff. The contribution of metals and other constituents to Koppers Pond from industrial process-related discharges has been reduced from historical levels because several of the past operations have ceased and no longer discharge to the Industrial Drainageway (Section 3.1.1.1). The effects of such reductions will be evaluated by comparisons in surface water quality and other measurements of changes in Koppers Pond from those observed during the Operable Unit 3 RI.

The other potential sources of COPCs to the pond relate to industrial and urban runoff, which could provide metals, PAHs, and other constituents.

The Old Horseheads Landfill is a potential ongoing source, although seeps from the Old Horseheads Landfill are not likely a significant source of COPCs to Koppers Pond.

Along the north shore of the pond, the landfill topography is not conducive to the formation of seeps, and the southern portion of the landfill (i.e., the portion proximal to and in contact with the pond) was reportedly filled with construction and demolition debris.

In addition, floc material formerly associated with treated waste water discharges to the Industrial Drainageway continues to be observed, albeit in reduced quantities. It is not known whether this floc is continuing to form, or whether it is flaking off from material formed in the past on the interior of the underground piping connecting Outfall 001T at the former Westinghouse Horseheads plant site to the Chemung Street Outfall.

4.2 COPCs IN AFFECTED ENVIRONMENTAL MEDIA

4.2.1 Surface Water

The available data suggest that COPCs are not present in Koppers Pond surface water at concentrations that could cause or contribute to unacceptable human health risk. The 1995 human health risk assessment, using data from a time frame in which treated industrial waste waters were being discharged to the Industrial Drainageway and then to Koppers Pond at rates higher than those at present, showed human health risks associated with surface water pathways less than 10^{-6}, the lower end of USEPA's target risk range.

There could be some potential for re-dissolution of COPCs from sediments into surface water as the chemistry of the pond adjusts to the reduction of past industrial waste water discharges. In addition, the effects (if any) of the observed floc associated with past discharges has not been fully evaluated.

Surface water data are not available to draw conclusions with respect to potential ecological risk. In prior sampling, certain metals were found at concentrations above applicable New York State Class C ambient water quality criteria (Table 9), but the current concentrations are not known. Accordingly, metals are considered a COPC for surface water in Koppers Pond. Also, even though the source is likely to be historic area-wide application, the pesticide compounds α-BHC and β-BHC cannot be ruled out at this time as COPCs for Koppers Pond surface water and potential contributors to ecological risks.

4.2.2 Pond Sediments

Based on the results of previous investigations, pond sediments have been the primary affected environmental medium at Koppers Pond. Metals and hydrophobic organic compounds such as PCBs, pesticides, and PAHs may adsorb onto sediments and potentially become available to the aquatic food web.

In the BHHRA, direct exposure to Koppers Pond sediments was not found to pose an unacceptable potential risk. The potential human health risk associated with consumption of fish taken from Koppers Pond was, however, estimated to be above USEPA's acceptable risk range. The majority of the potential incremental excess lifetime cancer risk is attributable to PCBs, although arsenic also contributes to the total potential cancer risk.

Ecological risk evaluations (CDM, February 1999; CEC, July 2003) have shown certain pesticides, metals, and PCBs in sediment potentially contributing to unacceptable ecological risk. The prediction of a potential risk based on the comparison of constituent concentrations in sediments to sediment screening benchmarks was not corroborated by whole sediment toxicity testing that showed no to very limited toxicity of sediments to aquatic organisms, nor are the concentrations in biota predicted by models consistent with actual measurements of COPC in aquatic organisms. Resolution of these issues will require further evaluation in the RI. The RI includes sampling and analysis of sediments to provide current data, but at present, pesticides, PCBs, and heavy metals are considered the likely COPCs for sediments in Koppers Pond. Although some PAHs have been detected in sediments, these appear to be of lesser consequence because of lower concentrations, less pervasive occurrence, and the absence of PAHs in fish tissue samples analyzed as part of the Operable Unit 3 RI.

4.3 Potential Human Receptors

Although local residents and trespassers entering Koppers Pond could be exposed via direct contact to the COPCs in sediment, such exposure is unlikely and, based on the human health risk assessment completed under Operable Unit 3, expected to be of little consequence. Shoreline surface soils and dried sediments could be contacted, but the pond bottom is mucky and not conducive to wading.

Anglers who catch and consume fish from Koppers Pond may also be exposed to COPCs in Koppers Pond. Risk evaluations based on currently available data suggest that, when conservative, default assumptions are employed, potential incremental excess lifetime cancer risks may exceed USEPA's 10^{-6} to 10^{-4} target risk range, and the HI for non-carcinogenic effects may exceed USEPA's target of 1.0. The RI includes additional fish tissue sampling to allow for a re-examination of potential human health risks based on current fish data.

4.4 POTENTIAL ECOLOGICAL RECEPTORS

Aquatic and semi-aquatic receptors may potentially be exposed to COPCs in pond sediment through direct exposure pathways (e.g., incidental ingestion of sediments) and food-chain pathways. Potential exposure pathways associated with the pond ecosystem are depicted on Figure 7. Species (i.e., receptors) that may potentially be exposed to COPCs, and expected to be included in the supplemental baseline ecological risk assessment (SBERA), are shown in Table 10 and are discussed in Section 6.6.2.

5.0 OBJECTIVES

This section describes the DQOs and specific RI/FS objectives.

5.1 DATA QUALITY OBJECTIVES

The DQOs related to the sampling program for the Site are as follows:

- The data will be gathered and developed in accordance with procedures appropriate for characterization and delineation of Site-related COPCs.
- The data will be developed using procedures and methods designed to resolve COPC concentrations at sufficiently low concentrations to allow meaningful comparisons to applicable and relevant risk screening concentrations.
- The data will be of known or acceptable precision, accuracy, and completeness, within the limits of the methods.

In developing the DQOs, a series of planning steps were conducted based on USEPA *Guidance on Systematic Planning using the Data Quality Objectives Process* ([QA/G-4] EPA/240/B-06/001, February 2006) to ensure that the type, quantity, and quality of environmental data are appropriate for their intended use. Acceptable precision, accuracy, and completeness guidelines are further described in the QAPP for the Site. DQOs have been designed to provide data regarding COPC concentrations in environmental media that are useable for human health and ecological risk assessment.

5.2 SPECIFIC RI/FS OBJECTIVES

The following paragraphs outline the specific objectives for the RI/FS for Koppers Pond. These objectives flow from the PCSM, review of the June 18, 2007 RI/FS Work Plan submittal, and discussions with USEPA; U.S. Department of the Interior, Fish and Wildlife Service (USFWS); and NYSDEC. Because the RI/FS is conducted in an iterative fashion, additional objectives may be developed during the performance of the RI/FS.

5.2.1 Remedial Investigation

The specific objectives of the RI are to update the existing surface water and sediment data to characterize the current Site conditions and fill data gaps remaining from prior Site studies as needed to characterize the Site sufficiently to allow assessments of potential human health and ecological risks associated with COPCs related to Koppers Pond. An additional objective is to update the understanding of the Site with respect to use by ecological receptors. A final, complementary objective is to identify at least one and ideally two reference ponds, in which, if needed, COPC concentrations in key media (i.e., sediments and fish) could be characterized in the future.

Physical and chemical data gaps that have been identified in the scoping of the RI include the following:

- Update existing surface water and sediment data to characterize current Site conditions and allow for a comparison of current conditions to those identified in the Operable Unit 3 RI sampling and other historical sampling events;
- Develop volume estimates and depth profiles of potentially impacted sediments currently in Koppers Pond;
- Characterize the hydrology of the pond, including surface water inflows, discharges, and residence time, and surface water-groundwater interaction; and
- Evaluate the potential for continuing sources of COPCs to surface water and sediment in Koppers Pond.

Data gaps related to potential continuing sources of COPCs include the following:

- Determine flow rates and COPC concentrations in discharges from ongoing manufacturing and other operations at the former Westinghouse Horseheads plant site;
- Document the occurrence and measure the COPC concentrations of any identified significant surface water discharges from commercial/industrial areas and any seeps associated with the Old Horseheads Landfill that discharge to Koppers Pond; and

- Document the occurrence and measure the COPC concentrations associated with any accumulations of floc material inside the underground piping leading to the Chemung Street Outfall.

In addition to these physical and chemical data, fish tissue sampling will also be conducted to facilitate evaluations of potential human health and ecological risks using current COPC concentration data. The fish tissue sampling will also allow for a comparison between COPC concentrations observed now and those observed during prior sampling events.

5.2.2 Baseline Human Health Risk Assessment

The objective of the BHHRA is to determine whether Site-related COPCs pose an unacceptable potential current or future risk to human health in the absence of remedial action. The baseline assessment will build on and update prior human health risk evaluations as needed, including the prior BHHRA (CDM, November 1995) with respect to updated constituent screening, exposure point concentrations, exposure scenarios and assumptions, pathways analysis, and toxicity factors.

5.2.3 Baseline Ecological Risk Assessment

The ecological risk assessment will update and supplement the earlier ecological risk evaluations as needed. The objectives of this task are to evaluate current and expected future conditions of Koppers Pond with respect to the following:

- Determine whether actual or potential ecological risks exist in the evaluated areas; and
- Provide adequate data and other information necessary for risk management decisions.

The ecological risk assessment will generally follow the step-wise process defined in ERAGS guidance, beginning with a revised SLERA to provide for updated evaluations and, as needed, a revised problem formulation for subsequent ecological risk studies (see discussion in Section 6.6.2).

5.2.4 Feasibility Study

If the updated BHHRA or BERA indicates that Site conditions pose a human health or ecological risk that constitutes a basis for a response action, an FS will be prepared to evaluate the alternative means available to remediate the Site conditions causing or contributing to such unacceptable risks. The outcome of the FS will be the identification of one or more technically feasible and effective alternatives, which, once implemented, will return the Site to a condition that does not pose unacceptable risk or exceed applicable or relevant and appropriate chemical standards.

6.0 RI/FS IMPLEMENTATION

Pursuant to the Settlement Agreement, Statement of Work, and USEPA guidance, the RI/FS includes the following tasks:

- Task 1 – RI/FS Work Plan,
- Task 2 – Community Relations,
- Task 3 – Site Characterization,
- Task 4 – Identification of Candidate Technologies,
- Task 5 – Treatability Studies,
- Task 6 – Baseline Risk Assessment,
- Task 7 – RI Report,
- Task 8 – Development and Screening of Remedial Alternatives, and
- Task 9 – FS Report.

The following paragraphs describe the work activities associated with each of these tasks. The SAP (including both the FSP and QAPP) and HASP provide specific procedural guidelines as to how these tasks will be performed.

6.1 TASK 1 – RI/FS WORK PLAN

This RI/FS Work Plan submittal represents substantial completion of Task 1 as defined in the Settlement Agreement and Statement of Work. As part of the development of this plan, the Group and its consultants have conducted the following activities:

- Initial scoping discussion with USEPA and USFWS representatives via conference call on December 13, 2006;
- Preparation and submittal of the PCSM on February 19, 2007 and follow-up on USEPA comments via conference call on April 5, 2007;
- Site inspection and tour with USEPA representatives on March 16, 2007;
- Site inspection focused on potential human health exposure pathways and ecological risk issues on May 16, 2007;

- Meeting by conference call with USEPA, USFWS, and NYSDEC representatives to review potential human health exposure pathways and ecological risk issues on May 23, 2007;

- Submittal of an RI/FS Work Plan on June 18, 2007 and review of USEPA preliminary comments on that plan; and

- Revising the prior RI/FS Work Plan submittal pursuant to the proposed resolution of issues at the October 11, 2007 meeting among the Group, USEPA, and other reviewers.

6.2 TASK 2 – COMMUNITY RELATIONS

Pursuant to Paragraph 27b of the Settlement Agreement, the Group has worked with the USEPA Remedial Project Manager and Community Involvement Coordinator to prepare a Site-specific Community Involvement Plan (CIP) for the Koppers Pond RI/FS. The completed CIP has been forwarded to NYSDEC, NYSDOH, and the public document repositories. Due to the nature of the work to be conducted for the RI, a Community Air Monitoring Plan is not required.

The Group and its consultants will continue to support USEPA, NYSDEC, and NYSDOH in their community relations efforts on an as-requested basis. This task will include preparation of a draft fact sheet for USEPA review announcing the initiation of RI field sampling activities. Support efforts may also include preparation of additional community notifications or other information for public dissemination and participation in public meetings, as necessary.

6.3 TASK 3 – SITE CHARACTERIZATION

Site characterization activities are designed to fill data gaps remaining from prior Site studies as needed to characterize the Site sufficiently to allow assessments of potential human health and ecological risks associated with Site-related COPCs in Koppers Pond.

6.3.1 Field Investigation

The purpose of the field investigation is to characterize the nature and extent of Site-related COPCs associated with Koppers Pond. A substantial amount of historical

information already exists related to Koppers Pond, as outlined in Section 3.0. As part of the RI, additional environmental sampling will be collected and analyzed as needed to supplement and update these existing data to reflect current conditions.

Field planning and support activities are focused on the assembly of the project team and subcontractors and confirming Site access. The Group has provided USEPA with the qualifications of its principal consultants and has finalized contractual arrangements. To conduct the RI, access will principally be needed to properties owned by the Village of Horseheads, Hardinge, and EWB (Figure 3). Access to other properties will be needed on a less-frequent basis. The Village of Horseheads and Hardinge are Respondents to the Settlement Agreement. The Group will arrange for access as needed with the remaining landowners. Based on prior experience at the Kentucky Avenue Wellfield Site, significant project delays associated with gaining needed property access for sampling are not anticipated.

The field investigation will include seven subtasks:

- Task 3.1 – Surveying and Mapping;
- Task 3.2 – Surface Water and Sediment Sampling;
- Task 3.3 – Pond Bathymetry;
- Task 3.4 – Assess Sediment Thickness;
- Task 3.5 – Assess Potential Ongoing Sources;
- Task 3.6 – Assess Pond Hydrology; and
- Task 3.7 – Fish Tissue Sampling.

Samples will be collected, handled, and shipped in accordance with the Site-specific SAP, and equipment decontamination and chain-of-custody procedures will be followed during sampling activities. The data generated during the investigation will be used to update and refine the previously conducted baseline human health and ecological risk assessments. The RI will be used to provide current Site information to evaluate potential remedial technologies.

As part of the above-listed field investigation tasks, local water bodies exhibiting physical characteristics similar to those of Koppers Pond, but not potentially affected by Site sources, will be identified as candidate reference ponds. Key media from the

reference ponds (i.e., sediments and fish) may be sampled in the future. The need for such sampling will be determined following comparison of the data collected as part of the work described in this Work Plan with the historical data. If the newly collected data indicate the absence of a potential risk, or that such potential risks are decreasing, collection of additional samples from either Koppers Pond or one or more reference ponds would likely not be needed. If the newly collected data from Koppers Pond suggest that COPCs in the pond may pose a risk, reference pond samples may be collected to determine whether such conditions are unique to Koppers Pond.

6.3.1.1 Task 3.1 – Surveying and Mapping

Survey control established for the Operable Unit 3 remediation of the Industrial Drainageway and USGS and other available local survey control will be reviewed and evaluated to determine if additional control is necessary for completion of the RI/FS tasks. If additional survey control is needed, such control will be established in the vicinity of Koppers Pond and tied to State Plane Coordinates and North American Vertical Datum of 1988.

Topographic mapping of the Koppers Pond area is available from Operable Unit 2 activities. This mapping is of suitable horizontal scale (1 inch = 50 feet) and contour interval (1 foot) for use as the base map for Operable Unit 4 field investigations. Figure 5 of this Work Plan was prepared using this available mapping. Field verification surveying will be conducted to ensure accurate tie-in to available survey control points.

A staff gauge will be installed in the pond to measure the pond surface elevation. The staff gauge will be surveyed to establish its reference elevation tied to Site control points. Staff gauge readings will be used for quality assurance and quality control (QA/QC) in the pond hydrology study (Section 6.3.1.6).

6.3.1.2 Task 3.2 – Surface Water and Sediment Sampling

Surface water and sediment samples will be collected from the pond and the outlet channel. The following locations are proposed for sample collection:

- Pond – 13 locations throughout the pond to provide sufficient coverage to allow comparisons to previously collected data and investigate the range of hydraulic conditions present in the pond (e.g., center channels, near-shore shallows); and

- Outlet Channels – One in the East Outlet, one in the West Outlet, and two in the downstream Outlet Channel.

The proposed sampling locations are shown on Figure 8. Pond sediments will be characterized vertically and the number of samples (estimated to be up to three per location) will be determined by the thickness of sediment. The sampling strategy is to collect a sample representative of the uppermost 6 inches of sediment and additional samples as needed to characterize each 12-inch increment of deeper sediments. Sampling increments will be determined from the visual inspection of the retrieved samples.

Surface water and sediment sampling procedures are described in Section 5.0 of the FSP. All data collected to date suggest that sediment thicknesses in the pond are less than about 21 to 24 inches. As described in the FSP, a contingency sampling methodology will be available and employed if sediments are thicker than 24 inches.

6.3.1.3 Task 3.3 – Pond Bathymetry

Cummings/Riter will measure and map bottom depths bathymetrically using hydrographic survey techniques linked to a global positioning system (GPS). To obtain the sediment depth measurements, Cummings/Riter will use a sampling boat outfitted with a GPS unit and an echo sounder that are integrated with a data logger. The echo sounder will be calibrated by using a portable depth gauge at two locations and adjusting the echo sounder to equate to the manual reading.

The boat will traverse the pond with depth soundings taken every 25 to 50 feet along the traveled route. Track lines will be monitored using GPS to ensure adequate spatial coverage of the pond. Horizontal positional accuracy will be approximately plus or minus one foot, and vertical accuracy will be approximately plus or minus 0.1 foot. Shallow areas (i.e., typically less than 2.5 feet) will be measured from the boat using a portable depth measurement tool. The manual depth measurement tool is simply a metal plate mounted on a handle that is pushed downward through the water until the sampler

feels resistance. Depth to sediment is measured by the length of submerged handle. Where portions of the pond are not navigable by boat (e.g., northwestern area), water depths will be estimated from shoreline observations or wading.

Upon completion of the field work, the digital data from the GPS and echo sounder will be processed and other field data will be compiled to develop a bathymetric map of the pond that is tied to survey control points.

6.3.1.4 Task 3.4 – Assess Sediment Thickness

Sediment thickness in the pond will be measured using a metal probe. The probe will be advanced into the sediment until refusal is reached on the sand and gravel deposits beneath the sediment. The depth of the sediment bottom will be used with the pond bottom data obtained from the bathymetric survey to estimate the sediment thickness and volume of sediment in the pond. Sediment thickness will be measured at points throughout the pond, including each of the sediment sampling locations shown on Figure 8.

The minimum number of thickness measurements is 13, corresponding to the proposed 13 sampling locations. Additional thickness measurements will be taken if the sediment thickness is found to be non-uniform. The sediment thickness data will also be used to determine how many sediment samples will be collected at each designated sampling location, as described in Section 6.3.1.2.

6.3.1.5 Task 3.5 – Assess Potential Ongoing Sources

There are several ongoing sources of potential impacts to Koppers Pond that will be investigated to assess if they are currently a source of impact to the pond. Figure 9 shows possible sources that are currently known. The following sections describe the tasks associated with assessing these potential sources.

Former Westinghouse Horseheads Plant: Surface water samples will be collected of the barrier well treated water discharge and the Cutler-Hammer discharge, both of which are located at the former Westinghouse Horseheads plant site (Figures 6 and 9). Both of these surface water samples will be analyzed for TCL and TAL analytes and for the general chemistry parameters ammonia, fluoride, hardness, nitrites, and total suspended

solids. Dissolved oxygen, pH, oxidation-reduction potential (ORP), temperature, and specific conductance will be determined in the field at the time of sampling. Also, if found to be present in any of the observed or sampled water courses, samples of the floc will also be collected for TCL and TAL analyses.

If flow meters are available, readings will be taken to determine discharge. Where flow meters are not available, discharges will be estimated from field observations.

In addition to this sampling, NYSDEC files will be reviewed and effluent data compiled with respect to recent discharge monitoring reports for the barrier well and Cutler-Hammer discharges.

Underground Discharge Pipe and Chemung Street Outfall: Treated discharges and storm water originating at the former Westinghouse Horseheads plant are conveyed to the Industrial Drainageway and ultimately to Koppers Pond via an underground pipe that terminates at the Chemung Street Outfall. A video survey of the pipe will be conducted between the plant and the outfall to gather information on alignment, floc accumulation, and potential sources other than the plant that tie into the pipe. The video survey will be used to assess whether floc material has accreted on the walls of the pipe and to locate other pipes that discharge into the main line leading to the Chemung Street Outfall.

NYSDOT will be contacted to retrieve available as-built information on the pipe leading from the former Westinghouse Horseheads plant site to the Chemung Street Outfall. Village of Horseheads records and the Southern Tier Central Regional Planning database will also be researched regarding storm sewer systems (i.e., inlets, catch basins, and underground piping) that contribute to the flow that emanates from the Chemung Street Outfall.

A surface water sample of the discharge of Chemung Street Outfall will be collected and analyzed for TCL and TAL analytes and for the general chemistry parameters ammonia, fluoride, hardness, nitrites, and total suspended solids. Dissolved oxygen, pH, ORP, temperature, and specific conductance will be determined in the field at the time of sampling. Also, if found to be present in any of the observed or sampled water courses, samples of the floc will also be collected for TCL and TAL analyses. If found to be

present in the Chemung Street Outfall discharge, a sample of the floc will also be collected for TCL and TAL analyses. Surface water flow at the time of sampling will be determined based on the depth of water in the discharge pipe and the estimated flow velocity.

Storm Water Runoff: Sources of significant storm water runoff that enter the underground discharge pipe upstream of the Chemung Street Outfall, the Industrial Drainageway downstream of the Chemung Street Outfall, or directly flow into Koppers Pond will be investigated. In this context, "significant" flows are those that contribute at least 10 percent of the flow in the receiving water course (as determined by contributory drainage area and runoff conditions) or which convey runoff from potential COPC sources (e.g., electrical substations).

A field reconnaissance will first be conducted of the study area to identify potential sampling locations, including storm water inflow points (e.g., catch basins, storm inlets), road culverts, culverts under the railroad, and runoff from the County DPW yard (i.e., road cinder and salt storage) and the Norfolk Southern railroad. Runoff pathways will be assessed by examination of available mapping and field reconnaissance focused on topography and channelization.

Samples of significant surface water inflows will then be collected based on flows, evidence of local staining, upstream potential sources (e.g., electrical substations), and results of any prior (Operable Unit 3) sampling. Surface water samples will be analyzed for TCL and TAL analytes and for the general chemistry parameters ammonia, fluoride, hardness, nitrites, and total suspended solids. Dissolved oxygen, pH, ORP, temperature, and specific conductance will be determined in the field at the time of sampling. Discharges will be estimated from field observations of channel geometry and flow velocity. If a targeted inflow sample location is found to by dry at the time of sampling, the field team will assess whether a sediment sample should be collected.

Landfill Seepage: The northern shore of the pond and the east bank in the lower reach of the Industrial Drainageway abut the Old Horseheads Landfill. A walkover survey of the shoreline will be conducted to inspect for physical evidence of seeps that may drain into

the pond (Figure 9). The topography along the north shore of the pond where it abuts the landfill is relatively flat and not conducive to the formation of seeps, and seeps are not expected in this area.

The east bank of the lower drainageway is steeper, and, if present, seeps are more likely to occur in this area. If seeps are found, they will be sampled, and flow rates will be estimated based on field observations. Seep samples will be analyzed for TCL and TAL analytes and for the general chemistry parameters ammonia, fluoride, hardness, nitrites, and total suspended solids. Dissolved oxygen, pH, ORP, temperature, and specific conductance will be determined in the field at the time of sampling.

6.3.1.6 Task 3.6 – Assess Pond Hydrology

The needed data will be compiled to examine the basic water balance of Koppers Pond. Readily retrievable information (e.g., precipitation runoff rates, flows from point-source discharges to the Industrial Drainageway, pond volume, flow in discharge channels) will be used to assess the pond water balance.

In addition, the hydrology of Koppers Pond will be studied to assess the interaction of local groundwater and surface water. With the discharges from the barrier wells and other surface waters entering the pond, it is anticipated that Koppers Pond typically recharges groundwater, although groundwater inflows may occur under certain conditions.

Groundwater and surface water elevations will be measured for a period of three months by installing transducers with data loggers in the pond and in existing groundwater monitoring wells proximate to Koppers Pond (i.e., CW-9S/9D, CW-10S/10D, and MW-112S). Well locations are shown on Figure 5. Manual measurements of pond surface water elevations using the staff gauge will be used for verification of the transducer readings.

The groundwater elevation data from this three-month study will then be compared to the 10+ years of groundwater level measurements collected as part of the Operable Unit 2 long-term monitoring program and other available data from prior studies. It is anticipated that the correlations between pond levels and groundwater levels from the

three-month study can then be extrapolated over the range of groundwater levels observed in long-term monitoring. This understanding of groundwater and surface water interaction will contribute to the model of COPC fate and transport.

6.3.1.7 Task 3.7 – Fish Tissue Sampling

The objective of the fish survey is to collect fish for evaluation of risks to human and ecological receptors. The fish collection goals are provided in the FSP (Section 4.7) and include collection of a range of species found in past sampling to be present in Koppers Pond. Such species include carp and other bottom-feeders as well as pelagic species. The supplemental fish sampling program will include collection of similar fish species to those collected in July 2003 for fillets (to support the BHHRA), as well as the collection of whole-body samples of fish that could be consumed by avian and mammalian piscivores to be evaluated in the SBERA.

Although it is difficult to ensure quantitatively the outcome of the pending fish collection efforts, the targets for these collections to provide relevant data for both risk assessments are as follows:

- Collect 10 individual carp of one size (10 to 13 inches);
- Collect 10 individual fish of similar-sized sunfish or crappie (8 to 10 inches); and
- Collect two types of composites of forage fish/minnows: three composites of smaller forage fish (30 to 100 millimeters [mm]) and three additional composites of larger forage fish/minnows (100 to 300 mm).

Fish sampling will be conducted with consideration of the sampling and processing protocols described in relevant USEPA and other guidance, including the following:

- *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories*, Volume 1 (USEPA, November 2000); and
- *Procedures for Collection and Preparation of Aquatic Biota for Contaminant Analysis* (NYSDEC, October 2002).

The survey will also be conducted in accordance with conditions set forth in the New York Scientific Collector's Permit.

Along with fish sampling, qualitative data of the available fish habitat will be collected by measuring the following:

- Assessment of in-pond cover (e.g., large woody debris, root wads, root mats, undercut banks, gravel bars, and macrophytes);
- Floodplain and land use around the pond; and
- Degree of canopy cover.

The fish habitat assessment is a qualitative tool to be used in conjunction with other data (e.g., surface water quality, sediment quality, fish examination) in the overall evaluation of the pond ecosystem and stressors that might affect populations of various fish species. This habitat assessment will also be used in conjunction with other data to develop an estimate of potential and sustainable fish populations and the yield of the pond for (human) edible fish.

6.3.2 DATA ANALYSIS

For the RI, samples collected from surface water and sediment will be analyzed for TCL VOCs, SVOCs, pesticides and PCBs, and TAL inorganic parameters. Sediment samples will also be analyzed for TOC, and selected samples for acid volatile sulfide/ simultaneously extracted metals (AVS/SEM). The AVS/SEM analyses will be performed on up to six of the 0- to 6-inch interval sediment samples, including the northwestern area of the pond (i.e., CDM Sample Location 13) where prior toxicity testing for the draft BERA indicated toxicity to *Hyalella azteca* (Section 3.3.3). AVS/SEM analyses can provide insights into the bioavailability of metals (USEPA, October 2001). Surface water samples collected for TAL metals will be analyzed for the total and dissolved fractions to facilitate direct comparisons to ambient water quality criteria.

Analyses will be conducted using USEPA methods. As described in Section 8.0 of the QAPP, the specific analytical methods to be employed include certain *USEPA SW-846*

methods, which have been selected, with USEPA concurrence, to achieve quantitation limits that are sufficiently low to allow for meaningful comparisons of analytical results to suitable ecological and human health risk screening criteria.

In addition, surface water samples will be analyzed for general chemistry parameters using USEPA methods. These general chemistry parameters and their rationale for selection are as follows:

- Ammonia and nitrites – potential stressors to fish and other aquatic organisms;
- Fluoride – indicator parameter previously associated with permitted waste water discharges from industrial operations located at the former Westinghouse Horseheads plant site;
- Hardness – parameter needed for the interpretation and comparison of certain metals concentrations to ambient water quality criteria; and
- Total suspended solids – parameter needed to evaluate total versus dissolved metals concentrations and possible indicator of sample disturbance (e.g., inclusion of sediment in seep sample).

Dissolved oxygen, pH, ORP, temperature, and specific conductance will be determined in the field at the time of surface water sample collection.

Sediments will likewise be characterized in the field for pH and ORP, and select sediment samples will be sent to a geotechnical testing laboratory for grain-size determination. Additionally, up to six sediment samples will be analyzed for AVS/SEM metals. The selection of sediment samples for grain-size determination and AVS/SEM analysis will be based on visual inspection of samples collected in the field with the objective of evaluating the range of sediment materials present in the pond and its outlet channels. It is anticipated that all or nearly all of the pond samples will contain predominantly silt and clay-sized materials, although there may be some coarsening with depth. The outlet channel samples are expected to exhibit a wider range of grain size.

Fish-tissue samples will be analyzed for TCL pesticides and PCBs, TAL metals, and lipid content. The collected fish will be examined in the field for any external signs of

deformities, tumors, or lesions. If such deformities are noted, equal numbers of individual fish showing the deformities, tumors, or lesions and individual fish without deformities will also be analyzed for SVOCs. The lipid content of fish samples will be determined to facilitate the evaluation of the concentrations of lipid-soluble constituents (e.g., PCBs).

For all analyses of COPC concentrations to be used in risk evaluations, the required laboratory data deliverable package will be prepared to provide the format and content consistent with data packages for Contract Laboratory Program (CLP) methods. These CLP-like data packages will be suitable for validation using the procedures specified by the National Functional Guidelines and the QAPP.

QA/QC samples, including trip blanks, duplicate samples, matrix spike/matrix spike duplicate samples, equipment blanks, and field blanks, will be collected at the frequencies defined in the Site QAPP. Table 11 summarizes the anticipated samples that will be collected and analyzed during the RI.

If submitted by USEPA, laboratories selected for analyses of project samples that are not certified under USEPA CLP will conduct project-specific performance evaluation samples. Such performance evaluations, if requested by USEPA, will be completed prior to the analytical laboratory being approved for use on the project.

6.3.3 Data Management Procedures

6.3.3.1 Analytical Support and Data Validation

DQOs that have been identified specify the quality of data required to support decisions regarding risk evaluations and potential remedial response activities. Analytical support will be provided by a qualified analytical laboratory, and data validation will be performed on approximately 20 percent of the analytical data. Data validation will be performed in accordance with National Functional Guidelines and the QAPP.

6.3.3.2 Data Evaluation/Geographic Information System Setup

Data evaluation includes the compilation and review of field and laboratory data generated during the RI field investigations. Wherever practicable, data assembly and

review will be assisted by using a geographic information system (GIS) to locate sample points and Site features. The GIS will require setup and customization for this Site including input of the historical analytical results into the database.

6.3.4 Site Characterization Summary

At the conclusion of the field investigation, a concise Site Characterization Summary Report will be prepared presenting the results of these studies and comparing these results to those from prior investigations. The Site Characterization Summary Report will provide the rationale and supporting documentation for inclusion or exclusion of data from previous investigations. This evaluation will be made in consideration of the following types of factors for specific analytes and environmental media:

- Analytes for which an ongoing source is identified versus those for which no ongoing or recent source is known;
- Quantitative comparability (e.g., relative percent difference) of sampling results for analytes from similar media in similar locations;
- Comparability of sampling, sample preparation, and analysis methods; and
- Tendency of the sampled medium to act as a reservoir of analytes (e.g., sediments) versus media in which COPC residency may be quite short (e.g., surface water).

The outcome from these evaluations will be the Site database to be used in subsequent risk assessments and, as needed, remedial action alternatives evaluations in the FS.

6.3.5 Fate and Transport Model Memorandum

The Statement of Work appended to the Settlement Agreement provides for a Fate and Transport Memorandum to be prepared, if requested by USEPA, following the completion of Site Characterization. If requested by USEPA, a Fate and Transport Model Memorandum will be prepared and submitted to present modeling of impacts to surface water and environmental receptors from sediments. The type of modeling to be performed would be reviewed with USEPA at the time of the request.

Fate and transport modeling using Site data may help to evaluate the migration potential of Site-related COPCs in surface water and sediments. Processes that may be analyzed, depending on the COPCs identified for the Site, include ion exchange, adsorption, oxidation/reduction for inorganic compounds, and biodegradation and bioaccumulation for organic compounds. Natural attenuation/recovery processes will also be evaluated.

6.4 TASK 4 – IDENTIFICATION OF CANDIDATE TECHNOLOGIES

An Identification of Candidate Technologies Memorandum will be prepared and submitted to assess, based on the information developed up to the time of preparation, the range of potentially viable technologies for remediation at Koppers Pond. Potential remedial technologies are expected to include the following:

- Access Restrictions:
  - Fencing and Security (including both current and potential future measures),
  - Institutional Controls (including the existing NYSDOH fish advisory);
- Monitoring and Maintenance:
  - Physical Monitoring,
  - Chemical Monitoring,
  - Maintenance;
- In-Situ Containment:
  - Subaqueous Capping;
- Sediment Removal and Disposal:
  - Dredging,
  - Dewatering,
  - On-Site Treatment/Disposal,
  - Off-Site Treatment/Disposal; and
- In Situ Treatment:
  - Physical Treatment,
  - Chemical Treatment,
  - Biological Treatment.

These remedial technologies cover the range of technologies required for analysis of a full range of remedial action alternatives, including the following:

- No Action;
- Monitored Natural Recovery;
- Enhanced Natural Recovery:
  - Hot Spot Removal,
  - In Situ Treatment;
- Partial Removal and Subaqueous Capping; and
- Sediment Removal and Off-Site Disposal.

6.5 TASK 5 – TREATABILITY STUDIES (AS NECESSARY)

The Statement of Work provides for the possibility of conducting treatability studies if needed to assist in the evaluation of potential remedial action alternatives. The need for and scope of any such studies will be developed in conjunction with the evaluation of the Site characterization data generated in the field investigation and the baseline human health and ecological risk assessments.

6.6 TASK 6 – BASELINE RISK ASSESSMENT

As discussed in Section 1.2 of this Work Plan, USEPA and the Group have recognized that much is known about the Site, such that, in negotiating the Settlement Agreement and the Statement of Work, it was envisioned that the RI would assess the extent to which the previously completed risk assessments would present findings that are representative of the current conditions in Koppers Pond. Therefore, the following discussions of the human health and ecological risk assessments primarily focus on the content of expected deliverables and the approach to assembling those deliverables. This approach recognizes the iterative and step-wise nature of the data evaluation and risk assessment process.

6.6.1 Human Health Risk Assessment

As an adjunct to the RI, a BHHRA will be conducted to determine whether Site-related COPCs pose a present or reasonably anticipated future potential risk to human health in the absence of any remedial action. Specifically, the BHHRA will focus on assessing potential risks to human health as a result of concentrations of COPCs in media of concern at the Site. It will characterize potential health risks via present or reasonably foreseeable future exposure pathways, addressing four major objectives:

- Identify the specific human receptor groups that may be exposed to COPCs present in media of concern;

- Characterize present and reasonably foreseeable future exposure pathways and scenarios relevant to the receptors that potentially may be exposed to any COPCs in such media;

- Characterize potential risks to human health associated with exposure to COPCs using available data for the media of concern; and

- For comparison, assess concentrations of COPCs at likely exposure points to applicable or suitably analogous Federal and State standards.

The BHHRA will be conducted in accordance with the Settlement Agreement and the Statement of Work appended thereto. Specifically, a Memorandum on Exposure Scenarios and Assumptions that describes the exposure scenarios and assumptions that will likely be employed in the BHHRA in light of the present and reasonably anticipated future land use of the Site will be prepared and submitted to USEPA within 30 days of USEPA approval of the Work Plan. The memorandum will contain, among other items, our current understanding of the conceptual site model, exposure routes of concern, and RAGS Part D Tables 1 and 4. If after review of the Memorandum on Exposure Scenarios and Assumptions, USEPA provides comments that result in modifications to the memorandum, a revised memorandum will be submitted to USEPA.

Within 45 days after the receipt of the last set of validated data, a Pathways Analysis Report (PAR) will be submitted to USEPA to describe the risk assessment process and detail how the risk assessment will be prepared. Building upon the Memorandum on Exposure Scenarios and Assumptions, the PAR will include completed RAGS Part D Tables 2, 3, 5, and 6, as described in the Statement of Work attached to the Settlement Agreement. If after review of the PAR, USEPA provides comments that result in modifications to the PAR, a revised PAR will be submitted to USEPA.

Following USEPA approval of the PAR, a BHHRA report will be submitted in draft form to USEPA for review and inclusion in the RI. The draft BHHRA will build upon the Memorandum on Exposure Scenarios and Assumptions, and the PAR and will include

completed RAGS Part D Tables 7 through 10, as described in the Statement of Work. If after review of the draft BHHRA, USEPA provides comments that result in modifications to the draft BHHRA, a revised BHHRA will be submitted to USEPA.

In developing the approach for the BHHRA, relevant USEPA guidance documents will be considered, including, but not limited to, the following:

- *Risk Assessment Guidance for Superfund (RAGS), Volume I - Human Health Evaluation Manual (Part A), Interim Final* (EPA-540-1-89-002)," OSWER Directive 9285.7-01A, December 1989;
- *Risk Assessment Guidance for Superfund (RAGS), Volume I - Human Health Evaluation Manual (Part D, Standardized Planning, Reporting, and Review of Superfund Risk Assessments), Final* (EPA 540-R-97-033), OSWER 9285.7-01D, December 2001;
- *Exposure Assessment Guidelines*, 1992a;
- *Guidance for Risk Characterization,* February 1995;
- *Risk Assessment Guidance for Superfund: Volume III, Part A, Process for Conducting Probabilistic Risk Assessment* (EPA 540-R-02-002), December 2001;
- *Risk Assessment Guidance for Superfund, Volume I: Human Health Evaluation Manual (Part E, Supplemental Guidance for Dermal Risk Assessment), Final* (EPA/540/R/99/005), July 2004;
- *Guidelines for Carcinogen Risk Assessment; Final* (EPA/630/P-03/001B), March 2005;
- *Supplemental Guidance for Assessing Susceptibility from Early-Life Exposure to Carcinogens* (EPA/630R-03/003F), March 2005;
- *Human Health Toxicity Values in Superfund Risk Assessments,* OSWER Directive 9282.7-53, December 2003;
- *Clarification to the 1994 Revised Interim Soil Lead Guidance for CERCLA Sites and RCRA Corrective Action Facilities,* OSWER Directive 9200.4-27, August 1998;

- *Revised Interim Soil Lead Guidance for CERCLA Sites and RCRA Corrective Action Facilities,* OSWER Directive 9355.4-12, July 1994;

- *User's Guide for the Integrated Exposure Uptake Biokinetic (IEUBK) Model for Lead in Children, (IEUBK) Windows © 32-bit version* (EPA 9285.7-42), May 2002;

- *Integrated Exposure Uptake Biokinetic (IEUBK) Model for Lead in Children, Windows© version* (IEUBK win 32 v1.0-252 build 264), December 2005;

- *Risk Assessment Guidance for Superfund: Volume I - Human Health Evaluation Manual: (Part B, Development of Risk-Based Preliminary Remediation Goals), Interim,* OSWER Directive 9285.7-01B, December 1991;

- *Human Health Evaluation Manual, Supplemental Guidance: Standard Default Exposure Factors,* OSWER Directive 9285.6-03, March 1991;

- *ProUCL Version 4.0 User Guide,* April 2007;

- *Exposure Factors Handbook, Volumes I, II, and III,* (EPA/600/P-95/002Fa,b,c), August 1997;

- *Integrated Risk Information System (IRIS),* http://www.epa.gov/iris/;

- *Recommendations of the Technical Review Workgroup for Lead for an Interim Approach to Assessing Risks Associated with Adult Exposures to Lead in Soil,* December 1996; and

- *Superfund Lead-Contaminated Residential Sites Handbook,* USEPA Lead Sites Workgroup, December 2002.

The BHHRA will be conducted in accordance with accepted risk assessment approaches and will include the hazard identification, dose response, exposure assessment, and risk characterization steps outlined by the National Academy of Sciences (1983).

6.6.2 Supplemental Baseline Ecological Risk Assessment

As an adjunct to the RI, potential risks to ecological receptors at Koppers Pond will be evaluated in an SBERA. The SBERA will fulfill two principal purposes:

- Determine whether actual or potential ecological risks exist in the evaluated areas; and

- Provide adequate data and other information necessary for risk management decisions.

This section of the Work Plan presents the general approach and guiding principles for evaluating potential risks to ecological receptors associated with Koppers Pond. This approach was developed in accordance with the following USEPA guidance documents:

- *Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments*, June 1997;

- *Guidelines for Ecological Risk Assessment*, May 1998;

- Framework for Ecological Risk Assessment, 1992b;

- *Developing a Work Scope for Ecological Assessments*, May 1992; and

- *Issuance of Final Guidance: Ecological Risk Assessment and Risk Management Principles for Superfund Sites*, October 1999.

USEPA has developed and issued detailed guidance for conducting ecological risk assessments. In 1992, USEPA (May 1992) presented a framework for ecological risk assessment that outlined the concepts of assessment and measurement endpoints. This guidance was followed by more comprehensive guidelines for ecological risk assessment (USEPA, May 1998), which placed emphasis on ensuring that the results of the assessment can be used to support risk management decisions. At about the same time, USEPA (June 1997) developed an eight-step process for performing ERAGS. The eight steps are listed below:

- Step 1: Preliminary Screening Level Assessment,
- Step 2: Screening Level Assessment,
- Step 3: Problem Formulation,
- Step 4: Study Design and Development of DQOs,
- Step 5: Verification of Field Sampling Design,
- Step 6: Site Investigation and Data Analysis,
- Step 7: Risk Characterization, and
- Step 8: Risk Management.

Many of the steps in the ERAGS process include Scientific Management Decision Points (SMDPs) for which the objectives include the following:

- Verify that the work conducted at each step is complete;
- Determine whether the risk assessment is proceeding in a direction that will support decision-making; and
- Determine the need, if any, for proceeding to the next step.

SMDPs provide an opportunity to fine tune and focus any additional ecological risk assessment activities to address the specific goals of the ecological risk assessment for the Study Area. For example, SMDPs provide the opportunity to exit the process where the weight of evidence indicates there is no need for further action. Consequently, not all eight of the ERAG steps are required for all Site evaluations. Alternatively, the outcome of the SMDPs may be the decision to take pre-emptive remedial action if one or more of the following apply:

- The estimated preliminary risks are great,
- The cost of cleanup is reasonable, or
- The likelihood of reaching a different conclusion via additional investigation is slim.

The USEPA Office of Solid Waste and Emergency Response (OSWER) has also issued a set of risk management principles that are relevant to ecological risk assessments and serve as a supplement to the ecological risk assessment guidance (USEPA, 1999a). This OSWER Directive recommends that the following key risk assessment/risk management questions be addressed at each SMDP:

- What ecological receptors should be protected?
- Is there an unacceptable ecological risk at the Site?
- Will the cleanup cause more ecological harm than current Site contamination?
- What cleanup levels are protective?

In addition to the above questions, the OSWER Directive identifies six principles that risk managers should address when scoping ecological risk assessments or when making ecological risk management decisions (USEPA, 1999a). The principles are as follows:

- Principle No. 1 - Reduce ecological risks to levels that will result in the recovery and maintenance of healthy local populations and communities of biota.
- Principle No. 2 - Coordinate with Federal, Tribal, and State Natural Resource Trustees.
- Principle No. 3 - Use Site-specific ecological risk data to support cleanup decisions.
- Principle No. 4 - Characterize Site risks.
- Principle No. 5 - Communicate risks to the public.
- Principle No. 6 - Remediate unacceptable ecological risks.

The principal objective of the ecological risk assessment is to evaluate the potential for adverse ecological effects to occur in local populations of biological receptors exposed to COPCs in the media (i.e., sediments, surface water, and prey items) from Koppers Pond and the two outlet streams (Figure 2). Sediment remediation of the Industrial Drainageway was completed by 2003 and, therefore, this area does not require additional assessment.

As mentioned in the PCSM, aquatic and semi-aquatic receptors may potentially be exposed to COPCs in pond sediment through direct exposure pathways (e.g., incidental ingestion of sediments) and food-chain pathways. Species (i.e., receptors) that may potentially be exposed to COPCs, and which are expected to be evaluated in the SBERA, include the following:

- ***Amphibians and Reptiles***: The species evaluated in the earlier draft BERA (CDM, February 1999), the green frog (*Rana clamitans melanota*) and painted turtle (*Chrysemys picta marginata*), will be re-evaluated to allow comparison of current potential risks to estimates of potential risk reported in the draft BERA.

- ***Fish***: The measured concentrations of COPCs in forage fish (minnows and/or young-of-year fish) will be used to evaluate potential risks to semi-aquatic upper trophic level receptors.

- ***Piscivorous Avian Species***: The SBERA will re-evaluate the potential risks to a great blue heron (*Ardea herodias*) (evaluated in the previous draft BERA) and also to a belted kingfisher (*Ceryle alcyon*). The kingfisher will be included to develop a range of potential risks to avian receptors.

- ***Piscivorous Mammalian Species***: The same species evaluated in the prior draft BERA (CDM, 1999), the mink (*Mustela vison*), will be re-evaluated in the SBERA. This evaluation will allow comparison of current potential risks to estimates of potential risk reported in the draft BERA. The SBERA will also include an evaluation of the suitability of Koppers Pond habitat for mink.

- ***Omnivorous Mammalian Species***: The same species evaluated in the prior draft BERA (CDM, February 1999), the raccoon (*Procyon lotor*), will be re-evaluated in the SBERA. This evaluation will allow comparison of current potential risks to estimates of potential risk reported in the draft BERA.

- ***Benthic Macroinvertebrates***: The draft BERA (CDM, February 1999) found no evidence of benthic toxicity in whole sediment toxicity tests using two sensitive test species. Nevertheless, the SBERA will, at a minimum, include a discussion of these results as well as a comparison of COPC concentrations in recently collected sediment to concentrations present when the draft BERA toxicity tests were conducted to determine whether the findings reported in the original draft BERA are sufficient to support developing risk management decisions.

Larger terrestrial herbivores (e.g., deer) or carnivores (e.g., fox), although reported in the area of Koppers Pond (CDM, February 1999), are not considered appropriate for further assessment in the SBERA because their habitats or prey base would not overlap significant portions of the pond, streams, and associated environments.

As described in the Statement of Work, the evaluation of potential ecological risks may be documented in as many as three interim and three final deliverables:

- SLERA;
- Final Revised SLERA, if requested by USEPA;
- SBERA Scope of Work;
- Final Revised SBERA Scope of Work, if requested by USEPA;
- Draft SBERA; and,
- Final SBERA Report.

Each of these is described in more detail below.

SLERA and Revised SLERA: The first ecological risk assessment-related deliverable, due to USEPA 45 days after the receipt of the last set of validated data is an SLERA. If after review of the SLERA, USEPA provides comments on the SLERA that result in modifications to the SLERA, a revised SLERA will be submitted to USEPA, as needed.

Consistent with Paragraph 27 of the Settlement Agreement, the SLERA conducted by USEPA in March 1996 for the Industrial Drainageway and Koppers Pond will be reviewed to determine the extent to which the evaluations described therein can be employed in the SLERA to be completed following receipt of all data resulting from the field activities described in this Work Plan.

SBERA Scope of Work and Revised SBERA Scope of Work: If after reviewing the Revised SLERA, USEPA determines that an SBERA is required, the third ecological risk assessment-related deliverable, due to USEPA within 30 days of USEPA's notification of the requirement of an SBERA, is an SBERA Scope of Work. If after review of the SBERA Scope of Work, USEPA provides comments on the SBERA Scope of Work that result in modifications to the SBERA Scope of Work, a revised SBERA Scope of Work will be submitted to USEPA, as needed.

Draft SBERA and Final SBERA Report: The fifth ecological risk assessment-related deliverable, due to USEPA within 60 days after the receipt of the last set of validated data (or 60 days after USEPA's approval of the SBERA Scope of Work, whichever is later), is a draft SBERA. If after review of the draft SBERA, USEPA provides comments on the draft SBERA that result in modifications to the draft SBERA, a revised SBERA will be submitted to USEPA, as needed.

6.7 TASK 7 – REMEDIAL INVESTIGATION REPORT

The data collected and evaluated in Tasks 3 through 6 will be presented in an RI report. The RI report will characterize Site-related COPCs and assess the potential risks to human health and the environment. The general outline for the RI report is as follows:

- Site background;
- Field investigation and technical approach, including field and analytical methodologies;
- Site description, including physical characteristics of the Site (e.g., climate, topography, hydrology, geology, hydrogeology, demography and land use, ecology);
- Nature and extent of COPCs, including sources, distribution, and trends;
- Fate and transport of COPCs;
- BHHRA;
- SBERA; and
- Summary and conclusions.

6.8 TASK 8 – DEVELOPMENT AND SCREENING OF REMEDIAL ALTERNATIVES

Remedial action objectives (RAOs) will be developed, and remedial alternatives will be screened as the initial step of the FS process to identify potential technologies that are applicable to the Site and could potentially achieve RAOs. The screening process will be conducted in accordance with applicable USEPA guidance and the NCP. The screening process will be documented in a Remedial Alternatives Screening Memorandum.

The remedial alternatives that survive the initial screening evaluation will be analyzed in more detail. Additionally, applicable or relevant and appropriate requirements (ARARs) will be identified and verified. The possible remedial alternatives will be compared to seven evaluation criteria, including:

- Compliance with ARARs;
- Overall protection of human health and the environment;

- Short-term effectiveness;
- Long-term effectiveness and permanence;
- Reduction of toxicity, mobility, or volume;
- Implementability; and
- Cost.

This detailed analysis of the individual alternatives and the comparative analysis among the alternatives will be conducted consistent with the NCP and applicable USEPA guidance.

6.9 TASK 9 – FEASIBILITY STUDY REPORT

After remedial alternatives have been screened and evaluated, an FS report will be prepared and will include the following:

- Introduction and Site background;
- FS objectives;
- Remedial action objectives;
- General response actions;
- Identification and screening of remedial technologies;
- Description and detailed analysis of remedial alternatives; and
- Summary and conclusions.

7.0 PROJECT SCHEDULE

Figure 10 shows the current schedule for completion of the RI/FS for Koppers Pond. This schedule is indexed to the Group's receipt of approval of the PCSM on April 26, 2007.

The schedule shown on Figure 10 is based on the current understanding of Site conditions and data requirements, and estimations of required agency review times. As shown on Figure 10, the total project duration is currently estimated to be approximately 28 months.

Several factors could impact the timing and sequencing of project tasks. Such factors include, but are not limited to, the following:

- Delays related to obtaining access agreements for off-Site sample locations;
- Adverse weather (a three-month tolling period is built into the current schedule based on the understanding of the timing of field work);
- Unanticipated subsurface and/or hazardous conditions; and
- Delays in receipt of Site-specific information or comments.

The schedule presented on Figure 10 provides for the conduct of treatability studies (Task 5), but these studies are not on the critical path and do not affect the overall project schedule.

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TABLES

CUMMINGS RITER

Table 1
Chronology of Plant Operations and Wastewater Discharges
Former Westinghouse Horseheads Plant Site

Year	Description
1952	Westinghouse begins manufacturing operations, with primary neutralization treatment of process wastewaters.
1957	Westinghouse upgrades wastewater treatment to include metals precipitation and clarification, and files discharge permit application with New York State Department of Health (NYSDOH) to address chromium, lead, and cyanide. Reported average flow of 2 million gallons per day (mgd).
1966	Westinghouse renews NYSDOH discharge permit. Reported average flow of 2 mgd.
1967	Westinghouse upgrades wastewater treatment plant to improve metals precipitation and solids separation (clarification) efficiency.
1969	Westinghouse renews NYSDOH discharge permit, which now includes monitoring requirements for cadmium, chromium, copper, lead, nickel, zinc, and other constituents.
1973	Westinghouse applies for and receives State Pollutant Discharge Elimination System (SPDES) permit with effluent limitations for copper, chromium, lead, and zinc. Discharge to Outfall 001W. Permit subsequently modified and renewed at various times with additional effluent limitations and monitoring requirements.
1985	Westinghouse Picture Tube Operations join with Toshiba to form TWEC.
1986	TWEC applies for and received separate SPDES permit with effluent limitations and monitoring requirements for cadmium, copper, chromium, lead, nickel, zinc, and other constituents with discharge to Outfall 001T.
1988	Westinghouse sells its Imaging and Sensing Technology Division to ISTC. ISTC conducts manufacturing operations at site and conveys wastewater to Westinghouse on-site treatment plant for subsequent discharge to Outfall 001W and to TWEC for discharge via Outfall 001T.
1989	In 1989, Westinghouse sells its interest in TWEC to Toshiba Corporation, which, subsequently conducts manufacturing operations at site as TDD and later MTPDA.
1990	TDD applies for and receives SPDES permit for its treated process wastewater discharges with discharge to Outfall 001T. Monitoring requirements and effluent limitations are set for aluminum, arsenic, chromium, copper, lead, nickel, silver, zinc, and other constituents. Permit subsequently modified and renewed at various times with additional effluent limitations and monitoring requirements.

CUMMINGS RITER

Table 1
Chronology of Plant Operations and Wastewater Discharges
Former Westinghouse Horseheads Plant Site

Year	Description
1994	Westinghouse sells its remaining Horseheads operations (i.e., manufacture of vacuum interrupters) to Cutler-Hammer. Cutler-Hammer conducts manufacturing operations at site and conveys wastewater to Westinghouse on-site treatment plant for subsequent discharge to Outfall 001W.
1995	Observation of whitish-brown floc first reported in Industrial Drainageway and Koppers Pond.
1996	Westinghouse closes on-site wastewater treatment plant. Outfall 001W relocated and new Outfall 001W receives only overflow from barrier well treatment facility. Westinghouse also separates boiler blowdown and compressor cooling water discharges from other discharges with discharge at new Outfall 002W. Cutler Hammer and ISTC plating wastewaters rerouted to Chemung County Sewer Authority. Other Cutler-Hammer and ISTC process wastewater and cooling water discharges routed to Cutler-Hammer Outfall 001CH (i.e., "old Outfall 001W) under a separate SPDES permit, although some ISTC discharges also routed to Outfall 001T.
2000	ISTC terminates its operations at the former Westinghouse Horseheads plant site.
2004	MTPDA terminates manufacturing operations and discharges of treated process wastewaters. Discharges from barrier well treatment system (which formerly provided process water to ongoing plant site operations) now predominantly sent for direct discharge to Industrial Drainageway via Outfall 001W. CBS terminates its (former Westinghouse) discharges of boiler blowdown, non-contact (compressor) cooling water, and deionized water wash discharges to Industrial Drainageway via its Outfall 002W. Only storm water runoff is discharged via Outfall 002W.
2007	CBS sells property to Silagi Development and Management, Inc., but retains use of Outfall 001W under SPDES Permit No. 0004103. Former Outfalls 001T and 002W are transferred to the new owner, but currently only storm water is discharged via these outfalls.

Table 2
Surface Water Sampling Locations and Field Characterization Data
Koppers Pond, Horseheads, New York

1994 and 1995 Operable Unit 3 RI Sampling								
Sample Location No.	Date of Sampling	Surface Water Characteristics						
		Water Depth (feet)	pH (s.u.)	Temperature		Conductivity		Dissolved Oxygen (mg/L)
				°C	°F	uS/cm	mv	
15	06/03/94	2.6	6.0	20.5	68.9	825	--	--
	06/06/95	2.5	7.6	28.2	82.8	--	646	5.2 @ 2.0 ft
16	06/02/94	2.2	6.0	21.0	69.8	550	--	--
	06/06/95	2.3	8.5	22.7	72.9	--	56	--
	06/08/95	--	5.7	--	--	--	217	4.4
	06/08/95	--	6.5	--	--	--	161	--
17	06/03/94	1.5	7.0	22.0				--
	06/06/95	2.8	--	29.3	84.7	--	--	1.0
	06/08/95	--	8.4	--	--	--	91	--
18	06/06/95	4.7	4.0	27.5	81.5	--	120	15.2 @ 3.5 ft
19	06/05/95	0.9	--	25.5	77.9	--	--	--
	06/08/95	--	8.9	--	--	--	93	9.9
20	06/05/95	1.3	--	23.5	74.3	--	--	--
	06/08/95	--	8.9	--	--	--	90	7.8
21	06/07/95	0.6	--	25.8	78.4	--	--	0.32
	06/07/95	--	8.1	--	--	--	96	--

See notes at end of table.

Table 2
Surface Water Sampling Locations and Field Characterization Data
Koppers Pond, Horseheads, New York

1998 CDM Sampling								
Sample Location No.	Depth of Sampling	Date of Sampling	Surface Water Characteristics					
			Water Depth (feet)	pH (s.u.)	Temperature		Conductivity (uS/cm)	Dissolved Oxygen (mg/L)
					°C	°F		
1	--	08/17/98	0.5	8.18	25.4	77.7	849	10.2
2	--	08/18/98	0.7	7.87	22.8	73.0	940	11.6
3	--	08/18/98	1.5	8.57	23.9	75.0	811	14.8
4	--	08/18/98	2.0	8.14	23.1	73.6	960	11.2
5	Surface	08/18/98	5.3	8.56	24.0	75.2	804	14.3
	Bottom			7.88	23.7	74.7	908	10.6
6	Surface	08/19/98	3.3	8.37	22.2	72.0	847	14.6
	Bottom			8.20	21.2	70.2	855	13.8
7	Surface	08/19/98	2.3	7.75	20.3	68.5	875	12.2
	Bottom			7.75	19.2	66.6	917	8.2
8	Surface	08/20/98	3.3	8.31	19.6	67.3	930	11.0
	Bottom			7.85	18.5	65.3	1,040	7.4
9	Surface	08/20/98	3.0	8.16	18.7	65.7	930	11.7
	Bottom			8.13	18.6	65.5	930	11.4
10	--	08/19/98	1.0	8.01	19.2	66.6	910	10.4
11	Surface	08/20/98	2.6	7.74	17.9	64.2	940	9.8
	Bottom			7.63	17.7	63.9	980	8.0
12	Surface	08/19/98	2.3	7.94	23.8	74.8	990	9.4
	Bottom			7.94	23.5	74.3	980	0.9
13	--	08/19/98	0.8	7.80	24.0	75.2	7,800	1.0

See notes at end of table.

Table 2
Surface Water Sampling Locations and Field Characterization Data
Koppers Pond, Horseheads, New York

2003 Sampling								
Sample Location No.	Depth of Sampling	Date of Sampling	Surface Water Characteristics					
			Water Depth (feet)	pH (s.u.)	Temperature		Conductivity (uS/cm)	Dissolved Oxygen (mg/L)
					°C	°F		
WQ-1	--	06/05/03	3.5	8.18	16.5	61.7	1,112	11.74
WQ-2	--	06/05/03	3.3	7.93	16.7	62.1	958	9.95
WQ-3	--	06/05/03	1.2	7.47	17.1	62.8	519	8.10
WQ-4	--	06/05/03	1.2	7.52	18.8	65.8	1,069	7.83

Notes:

1. *Operable Unit 3 RI data from Philip Environmental (March 1996). 1998 data from CDM (February 1999). 2003 data from CEC (July 2003).*
2. *For sampling locations, see Figure 3.*
3. *"--" indicates data not reported.*

Table 3
Historical Surface Water Analytical Data
Koppers Pond, Horseheads, New York

Parameter	Concentration (ug/L) by Sample Location and Sampling Date					
	SW-15 06/03/94	SW-16 06/02/94	SW-17 06/03/94	SW-18 06/02/94	SW-19 06/01/94	SW-20 06/01/94
Volatile Organic Compounds						
4-Methyl-2-penanone	10 U	10 U	10 UJ	10 U	**1 J**	**5 J**
Trichloroethylene	**3 J**	**2 J**	10 U	**2 J**	10 U	**3 J**
Pesticides and PCBs						
Alpha-BHC	0.05 U	**0.17 P**	0.05 U	**0.20 P**	**0.22 P**	0.05 U
Beta-BHC	0.05 U	0.05 U	0.05 U	0.05 U	0.05 U	**0.28 P**
Irnorganics						
Aluminum	90 U	**264**	**113 B**	**169 B**	**187 B**	**449**
Antimony	6.0 U	6.0 U	6.0 U	6.0 U	6.0 U	6.0 U
Arsenic	3.0 U	3.0 U	3.0 U	**3.0 JB**	**3.0 JB**	3.0 U
Barium	**249 J**	**238**	**235 J**	**224**	**223 J**	**239 J**
Cadmium	5.0 U	5.0 U	5.0 U	5.0 U	5.0 U	5.0 U
Calcium	R	R	R	R	R	R
Chromium	R	10 U	10 U	10 U	10 U	10 U
Copper	R	10 U	10 U	10 U	10 U	**12.0 R**
Iron	R	R	R	R	R	R
Lead	R	R	R	R	R	R
Magnesium	R	R	R	R	R	R
Manganese	R	R	R	R	R	R
Potassium	**5,360 J**	**6,260 J**	**5,640 J**	**5,380**	**5,350**	**5,670 J**
Sodium	R	R	R	R	R	R
Zinc	R	R	R	R	R	R
Fluoride	**7,000**	NA	**3,700**	NA	**3,700**	**3,700**
Total Suspended Solids	NA	NA	NA	NA	NA	NA
Hardness	NA	NA	NA	NA	NA	NA

See notes at end of table.

Table 3
Historical Surface Water Analytical Data
Koppers Pond, Horseheads, New York

Parameter	Concentration (ug/L) by Sample Location and Sampling Date						
	SW-15B 06/06/95	SW-16B 06/08/95	SW-17B 06/06/95	SW-18B 06/06/95	SW-19B 06/05/95	SW-20B 06/05/95	SW-21B 06/07/95
Volatile Organic Compounds							
4-Methyl-2-penanone	NA	NA	NA	NA	NA	NA	NA
Trichloroethylene	NA	NA	NA	NA	NA	NA	NA
Pesticides and PCBs							
Alpha-BHC	NA	NA	NA	NA	NA	NA	NA
Beta-BHC	NA	NA	NA	NA	NA	NA	NA
Irnorganics							
Aluminum	92 U	588	92 U	92 U	92 U	118	324
Antimony	8.84	7.14	9.27	9.91	8.63	7.99	14.8
Arsenic	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U
Barium	250	209	179	172	171	192	310
Cadmium	3.0 U	3.0 U	3.0 U	3.0 U	3.0 U	3.0 U	20
Calcium	86,000	68,200	54,000	54,000	56,600	66,900	102,000
Chromium	10.7 J	8.0 UJ	8.0 UJ	8.0 UJ	8.0 U	8.0 U	28.5
Copper	23 U	23 U	23 U	23 U	23 U	23 U	35.7
Iron	83.2	1,130	109	56.1	102	359	689
Lead	33.0	41.6	42.4	36.8	43.0	48.6	345
Magnesium	13,700	11,800	13,000	13,000	12,400	13,400	14,800
Manganese	14.8	36.0	15.7	13.2	18.1 J	24.3 J	17.8 J
Potassium	4,320	3,140	3,800	3,930	3,670	4,190	4,580
Sodium	60,200	59,200	62,300	63,900	58,500	59,400	70,200
Zinc	42.6	64.4	44.0	36.9	48.2	59.0	189
Fluoride	NA	NA	NA	NA	NA	NA	NA
Total Suspended Solids	5,000 U	5,000 U	46,000	5,000 U	31,900	35,300	35,300
Hardness	302,000	266,000	217,000	213,000	216,000	287,000	287,000

See notes at end of table.

Table 3
Historical Surface Water Analytical Data
Koppers Pond, Horseheads, New York

1. *Data from Philip Environmental (March 1996).*
2. *Only analytes detected in one or more samples are listed. Other analytes (e.g., SVOCs, PCBs) were not detected in any surface water sample above reporting limits.*
3. *For sampling locations, see Figure 3.*
4. *For clarity, all detections are shown in* ***bold-face type****.*
5. *"NA" indicates sample not analyzed for this constituent.*
6. *Organic data qualifiers:*
   - *U - not detected at indicated detection limit*
   - *J - analyte detected, but concentration is an estimated value because the result is less than the quantitation limit or quality control criteria were not met.*
   - *P - percent difference between results from both columns was greater than 25 percent.*
7. *Inorganic data qualifiers:*
   - *U - not detected at indicated detection limit*
   - *B - detected concentration below quantitation limit but above instrument detection limit.*
   - *J - constituent also detected in corresponding method blank*
   - *R - data rejected in validation.*

Table 4
Historical Sediment Sampling Data - Inorganics
Koppers Pond, Horseheads, New York

Parameter	Concentration (mg/kg) by Sample Location and Sampling Date						
	SD-15 06/03/94	SD-16 06/02/94	SD-17 06/03/94	SD-18 06/02/94	SD-19 06/01/94	SD-20 06/01/94	SD-15B 06/06/95
Aluminum	11,100	13,300	15,000	8,590	10,400	9,920	NA
Antimony	9.5 UJ	7.1 UJ	10.9 UJ	7.2 UJ	7.1 UJ	7.1 UJ	NA
Arsenic	1.9 J	7.2 J	10.9 UJ	4.3 J	7.5 UJ	5.5 J	NA
Barium	361	239	442	224	164	137	NA
Beryllium	0.95 U	0.71 U	1.1 U	0.72 U	0.71 U	1.0 B	NA
Cadmium	125 J	1.2 U	1.8 U	3.1	1.2 U	1.2 UJ	549 J
Calcium	33,200	2,440	15,700	17,200	8,330	6,530	NA
Chromium	151 J	18.8	63.1 J	39.3 J	19.0	17.1 J	357 J
Cobalt	11.1 B	12.7	17.1 B	10.3 B	14.5	11.7 B	NA
Copper	247 J	16.6	59.1 J	60.8 J	17.4	19.9 J	NA
Iron	21,200	30,800	38,100	23,500	28,300	23,400	NA
Lead	93	10.5 J	33.8 J	12.8 J	12.7 J	15.8 J	148 J
Magnesium	3,880	3,850	5,900	4,400	3,670	4,580	NA
Manganese	137 J	721 J	1,470 J	337 J	421 J	448 J	NA
Mercury	0.51	0.12 U	0.16 U	0.12 U	0.11 U	0.11 U	1.53 J
Nickel	125	26.5	80.6	43.8	25.3	24.2	NA
Potassium	1,220	525 J	1,370 J	791 J	577 J	513 J	NA
Selenium	0.95 U	0.68 UJ	1.10 U	0.72 UJ	0.75 UJ	0.74 UJ	NA
Silver	6.7 J	0.05 U	3.60 UJ	0.05 U	0.05 U	0.05 U	NA
Sodium	479 B	304 B	445 B	293 B	285 B	274 B	NA
Thallium	1.9 U	1.4 U	2.2 UJ	1.4 UJ	1.5 U	1.5 U	NA
Vanadium	33.7 J	20.4 J	28.3 J	14.7	17.7	14.8	NA
Zinc	1,000 J	79.9 J	197 J	160 J	72.1 J	96.3 J	NA
Total Cyanide	R	R	R	R	R	R	NA

See notes at end of table .

Table 4
Historical Sediment Sampling Data - Inorganics
Koppers Pond, Horseheads, New York

Parameter	Concentration (mg/kg) by Sample Location and Sampling Date						
	SD-16B 06/08/95	SD-17B 06/06/95	SD-18B 06/06/95	SD-19B 06/05/95	SD-20B 06/05/95	SD-01 08/17/98	SD-02 08/18/98
Aluminum	NA	NA	NA	NA	NA	8,460	7,730 J
Antimony	NA	NA	NA	NA	NA	1.10 B	6.2 BJ
Arsenic	NA	NA	NA	NA	NA	3.60	5.7 BJ
Barium	NA	NA	NA	NA	NA	203	536 J
Beryllium	NA	NA	NA	NA	NA	0.72 B	0.91 BJ
Cadmium	13.2	2.24 J	52.5 J	28.9 J	29.9 J	7.0	54.1 J
Calcium	NA	NA	NA	NA	NA	16,000 *	146,000 *J
Chromium	36.5 J	35.4	189 J	97.6 J	58.8 J	40.7	159 J
Cobalt	NA	NA	NA	NA	NA	8.4 B	7.9 BJ
Copper	NA	NA	NA	NA	NA	34.9	176 J
Iron	NA	NA	NA	NA	NA	18,900	12,600 J
Lead	101	31.5	102 J	164 J	159 J	91.6	393 J
Magnesium	NA	NA	NA	NA	NA	3,480	4,370 BJ
Manganese	NA	NA	NA	NA	NA	261	115 J
Mercury	0.120	0.122	0.877 J	0.181 J	0.148 J	0.08 UJ	0.25 UJ
Nickel	NA	NA	NA	NA	NA	27.7	97.3 J
Potassium	NA	NA	NA	NA	NA	428 B	764 BJ
Selenium	NA	NA	NA	NA	NA	1.5 BNJ	3.8 BNJ
Silver	NA	NA	NA	NA	NA	0.72 B	6.4 BJ
Sodium	NA	NA	NA	NA	NA	237 B	658 BJ
Thallium	NA	NA	NA	NA	NA	1.2 UJ	3.8 UJ
Vanadium	NA	NA	NA	NA	NA	12.3 B	9.5 BJ
Zinc	NA	NA	NA	NA	NA	216	1,010 J
Total Cyanide	NA	NA	NA	NA	NA	0.29 U	0.35 UJ

See notes at end of table .

Table 4
Historical Sediment Sampling Data - Inorganics
Koppers Pond, Horseheads, New York

Parameter	Concentration (mg/kg) by Sample Location and Sampling Date						
	SD-03 08/18/98	SD-04 08/18/98	SD-05 08/18/98	SD-06 08/19/98	SD-07 08/19/98	SD-08 08/20/98	SD-08 (0-3) 08/20/98
Aluminum	9,730 J	5,850	6,760 J	4,840 J	5,040 J	5,850 EJ	7,380 EJ
Antimony	1.8 BJ	0.95 B	14.5 BJ	6.3 BJ	1.6 UJ	3.2 BNJ	11.5 BNJ
Arsenic	5.3 J	4.9	4.1 UJ	4.8 BJ	1.9 UJ	2.7 UNJ	3.7 UNJ
Barium	251 J	77.9	565 J	510 J	346 J	393 ENJ	739 ENJ
Beryllium	0.6 BJ	0.48 B	0.87 BJ	0.76 BJ	0.45 BJ	0.16 UNJ	0.48 BNJ
Cadmium	9 J	1.3 B	52.8 J	59.9 J	82 J	238 EJ	214 EJ
Calcium	18,100 *J	31,600 *	147,000 *J	175,000 *J	63,400 *J	77,400 EJ	180,000 EJ
Chromium	34.9 J	22.7	142 J	164 J	98 J	164 EJ	197 EJ
Cobalt	7.9 BJ	5.9 B	6.2 BJ	7.3 BJ	7.7 J	7.1 BNJ	7.1 BNJ
Copper	32.7 J	19.5	135 J	179 J	130 J	282 EJ	294 EJ
Iron	19,200 J	15,100	12,500 J	9,630 J	9,860 J	7,850 EJ	10,700 EJ
Lead	93.1 J	31.2	532 J	427 J	234 J	355 EJ	617 EJ
Magnesium	2,660 J	4,810	5,670 BJ	3,900 BJ	2,250 J	2,410 BEJ	3,730 BEJ
Manganese	245 J	288	126 J	101 J	96.5 J	69 ENJ	111 ENJ
Mercury	0.10 UJ	0.07 UJ	0.29 UJ	0.23 UJ	0.12 UJ	1.2 *J	0.74 *J
Nickel	22.7 J	18.5	60.5 J	90.1 J	90.8 J	R	R
Potassium	629 BJ	366 B	702 BJ	513 BJ	415 BJ	742 BEJ	1,080 BEJ
Selenium	1.5 UNJ	1.0 UNJ	4.4 UNJ	3.3 UNJ	2.0 UNJ	2.9 UNJ	3.9 UNJ
Silver	1.1 BJ	0.28 U	6.9 BJ	6.7 BJ	4.5 BJ	11.4 J	13.7 J
Sodium	244 BJ	160 B	955 BJ	809 BJ	442 BJ	517 BJ	945 BJ
Thallium	1.5 UJ	1.1 B	4.5 UJ	3.4 UJ	2.1 UJ	3.0 UNJ	4 UNJ
Vanadium	14.2 BJ	10.0 B	9.7 BJ	6.4 BJ	6.7 BJ	11.6 BNJ	10.6 BNJ
Zinc	244 J	101	1,130 J	1,020 J	1,300 J	3,500 EJ	3,400 EJ
Total Cyanide	0.14 UJ	0.10 U	0.56 BJ	0.32 UJ	0.19 UJ	1.2 BJ	347 J

See notes at end of table .

Table 4
Historical Sediment Sampling Data - Inorganics
Koppers Pond, Horseheads, New York

Parameter	Concentration (mg/kg) by Sample Location and Sampling Date						
	SD-08 (3-9) 08/20/98	SD-08 (9-12) 08/20/98	SD-08 (12-17) 08/20/98	SD-09 08/20/98	SD-09 (0-6) 08/20/98	SD-09 (6-12) 08/20/98	SD-10 08/19/98
Aluminum	8,860 EJ	12,600 J	20,500 EJ	10,000 EJ	NA	NA	9,900 J
Antimony	2.0 UNJ	2.0 UJ	1.3 UNJ	3.8 BNJ	NA	NA	NA
Arsenic	3.9 BNJ	6.0 BJ	4.4 NJ	NA	NA	NA	NA
Barium	546 ENJ	340 J	306 ENJ	558 ENJ	NA	NA	473 J
Beryllium	0.24 BNJ	0.83 BJ	1.1 BNJ	0.27 BNJ	NA	NA	NA
Cadmium	418 EJ	34 J	0.84 BEJ	304 EJ	NA	NA	135 J
Calcium	115,000 EJ	21,900 *J	6,580 EJ	NA	NA	NA	NA
Chromium	311 EJ	140 J	R	231 EJ	NA	NA	329 J
Cobalt	13.2 BNJ	12.2 BJ	8.0 BNJ	10.8 BNJ	NA	NA	10.1 BJ
Copper	570 EJ	207 J	37.1 EJ	371 EJ	NA	NA	354 J
Iron	12,700 EJ	19,100 J	18,500 EJ	NA	NA	NA	NA
Lead	378 EJ	52.9 J	18.8 EJ	509 EJ	NA	NA	459 J
Magnesium	3,750 EJ	3,480 J	3,770 EJ	NA	NA	NA	NA
Manganese	100 ENJ	122 J	123 ENJ	NA	NA	NA	NA
Mercury	1.2 *J	0.07 UJ	0.44 *J	0.38 *J	NA	NA	NA
Nickel	210 NJ	R	R	NA	NA	NA	156 J
Potassium	975 BEJ	1,060 BJ	1,460 BEJ	NA	NA	NA	NA
Selenium	2.5 UNJ	3.6 NJ	1.7 BJ	NA	NA	NA	NA
Silver	16.1 J	4.5 BJ	0.42 UNJ	15.6 J	NA	NA	9.3 BJ
Sodium	522 BJ	499 BJ	339 BJ	NA	NA	NA	NA
Thallium	2.6 UNJ	2.5 UJ	1.6 UNJ	NA	NA	NA	NA
Vanadium	17.3 BNJ	18.6 BJ	23.3 NJ	18 BNJ	NA	NA	12.1 BJ
Zinc	5,450 EJ	369 J	R	4,470 EJ	NA	NA	2,120 J
Total Cyanide	0.94 BJ	0.23 UJ	1.5 J	0.5 BJ	NA	NA	NA

See notes at end of table .

Table 4
Historical Sediment Sampling Data - Inorganics
Koppers Pond, Horseheads, New York

Parameter	Concentration (mg/kg) by Sample Location and Sampling Date						
	SD-11 08/20/98	SD-12 (0-6) 08/19/98	SD-12 (6-12) 08/19/98	SD-12 (12-18) 08/19/98	SD-12 (18-21) 08/19/98	SD-13 08/19/98	SD-20 8/201998
Aluminum	NA	8,320 J	5,480 J	4,600 J	11,300 J	6,180 J	7,300 EJ
Antimony	NA	3.1 BJ	2.9 BJ	2.7 BJ	2.2 BJ	10.4 BJ	3.6 BNJ
Arsenic	NA	6.1 BJ	4.1 BJ	5.4 BJ	5.4 BJ	7.8 BJ	4.3 BNJ
Barium	NA	680 J	485 J	326 J	1,490 J	684 J	522 ENJ
Beryllium	NA	0.6 BJ	0.41 BJ	0.6 BJ	1.1 BJ	0.47 BJ	0.15 UNJ
Cadmium	NA	583 J	647 J	749 J	44.9 J	415 J	502 EJ
Calcium	NA	128,000 *J	120,000 *J	133,000 *J	25200 *J	125,000 *J	110,000 EJ
Chromium	NA	330 J	245 J	460 J	144 J	342 J	246 EJ
Cobalt	NA	10.4 BJ	10.4 BJ	18.6 BJ	13.5 BJ	7.2 BJ	8.6 BNJ
Copper	NA	694 J	680 J	960 J	212 J	544 J	541 EJ
Iron	NA	11,700 J	9,230 J	11,400 J	16700 J	10,700 J	9,240 EJ
Lead	NA	1440 J	729 J	349 J	61 J	2210 J	734 EJ
Magnesium	NA	4,220 J	3,490 J	4,120 J	3280 J	3,690 BJ	3,520 BEJ
Manganese	NA	109 J	82 J	97 J	118 J	99 J	84 ENJ
Mercury	NA	0.64 J	0.17 UJ	0.77 J	0.40 J	0.23 UJ	1.0 J
Nickel	NA	142 J	143 J	395 J	147 J	155 J	R
Potassium	NA	780 BJ	438 BJ	404 BJ	997 BJ	569 BJ	1,100 BEJ
Selenium	NA	2.9 UNJ	2.6 UNJ	2.1 UNJ	3.1 NJ	3.4 UNJ	2.7 UJ
Silver	NA	38 J	27.5 J	23.1 J	6.3 J	39.6 J	25.6 J
Sodium	NA	744 BJ	589 BJ	576 BJ	604 BJ	863 BJ	633 BJ
Thallium	NA	3.0 UJ	2.6 UJ	2.1 UJ	2.5 BJ	3.5 UJ	2.8 UNJ
Vanadium	NA	11.3 BJ	7.7 BJ	10.5 BJ	16 BJ	8.7 BJ	22.1 BNJ
Zinc	NA	12,500 J	12,300 J	9,690 J	357 J	6,820 J	6,680 EJ
Total Cyanide	NA	0.27 UJ	0.24 UJ	0.2 UJ	0.36 BJ	0.33 UJ	0.26 UJ

See notes at end of table .

Table 4
Historical Sediment Analytical Data
Koppers Pond, Horseheads, New York

1. *Operable Unit 3 RI data are from Philip Environmental (March 1996).*
   1998 data are from CDM (February 1999).
2. *For sampling locations, see Figure 3.*
3. *For clarity, all detections are shown in* ***bold-face type*** *.*
4. *"NA" indicates data not available due to missing page in CDM (February 1999) report.*
5. *All results are reported on a dry-weight basis.*
6. *Inorganic data qualifiers:*
   U - not detected at indicated reporting limit.
   B - detected concentration below quantitation limit but above instrument detection limit.
   N - matrix spike/matrix spike duplicate (MS/MSD) recoveries outside control limits.
   J - constituent also detected in corresponding method blank.
   ** - the relative percent difference (RPD) of the MS/MSD recovered outside control limits.*
   E- reported concentration is estimated due to matrix interference.
   R - data rejected in validation.

Table 5
Historical Sediment Analytical Data - VOCs and SVOCs
Koppers Pond, Horseheads, New York

Parameter	Concentration (ug/kg) by Sample Location and Sampling Date						
	SD-15 06/03/94	SD-16 06/02/94	SD-17 06/03/94	SD-18 06/02/94	SD-19 06/01/94	SD-20 06/01/94	SD-15B 06/06/95
Volatile Organic Compounds							
Carbon disulfide	**7 J**	12 UJ	**5 J**	13 UJ	12 U	12 U	NA
Methylene chloride	**45 BJ**	38 UJ	43 UJ	34 UJ	15 J	12 UJ	NA
Toluene	15 U	12 U	15 U	13 U	12 U	**5 J**	NA
Semi-Volatile Organic Compounds							
Acenaphthene	490 U	400 U	**210 J**	420 U	400 U	390 U	891 UJ
Anthracene	490 U	400 U	**170 J**	420 U	400 U	390 U	891 UJ
Benzo(a)anthracene	**33 J**	400 U	**140 J**	420 U	**23 J**	390 U	891 UJ
Benzo(a)pyrene	**47 J**	400 U	**110 J**	420 U	400 U	390 U	**200 J**
Benzo(b)fluoranthene	**40 J**	400 U	**150 J**	420 U	400 U	390 U	**350 J**
Benzo(ghi)perylene	**28 J**	400 U	**68 J**	420 U	400 U	390 U	**200 J**
Benzo(k)fluoranthene	**44 J**	400 U	**180 J**	420 U	400 U	390 U	891 UJ
bis(2-Ethylhexyl) phthalate	**100 J**	400 U	**35 J**	**29 J**	400 U	**29 J**	891 UJ
Carbazole	490 U	400 U	**40 J**	420 U	400 U	390 U	NA
Chrysene	**58 J**	400 U	**260 J**	**22 J**	400 U	390 U	**280 J**
Dibenzofuran	490 U	400 U	**140 J**	420 U	400 U	390 U	891 UJ
Di-n-butyl phthalate	**68 J**	**22 J**	490 U	**26 J**	400 U	390 U	891 UJ
Fluoranthene	**100 J**	400 U	**740**	**33 J**	400 U	390 U	**530 J**
Fluorene	490 U	400 U	**250 J**	420 U	400 U	390 U	891 UJ
Indeno(1,2,3-cd)pyrene	490 U	400 U	**61 J**	420 U	400 U	390 U	**130 J**
Naphthalene	490 U	400 U	**36 J**	420 U	400 U	390 U	891 UJ
Phenanthrene	**43 J**	400 U	**190 J**	420 U	400 U	390 U	891 UJ
Pyrene	**63 J**	400 U	**440 J**	**25 J**	400 U	390 U	891 UJ

See notes at end of table.

Table 5
Historical Sediment Analytical Data - VOCs and SVOCs
Koppers Pond, Horseheads, New York

Parameter	Concentration (ug/kg) by Sample Location and Sampling Date				
	SD-16B 06/08/95	**SD-17B 06/06/95**	**SD-18B 06/06/95**	**SD-19B 06/05/95**	**SD-20B 06/05/95**
Volatile Organic Compounds					
Carbon disulfide	NA	NA	NA	NA	NA
Methylene chloride	NA	NA	NA	NA	NA
Toluene	NA	NA	NA	NA	NA
Semi-Volatile Organic Compounds					
Acenaphthene	597 U	559 U	917 U	1,270 U	750 U
Anthracene	597 U	559 U	917 U	1,270 U	750 U
Benzo(a)anthracene	597 U	559 U	917 U	1,270 U	**300 J**
Benzo(a)pyrene	**87 J**	559 U	917 U	**190 J**	**300 J**
Benzo(b)fluoranthene	**170 J**	559 U	**170 J**	**400 J**	**520 J**
Benzo(ghi)perylene	**69 J**	559 U	917 U	**160 J**	**230 J**
Benzo(k)fluoranthene	597 U	**90 J**	917 U	1,270 U	750 U
bis(2-Ethylhexyl) phthalate	597 U	559 U	**110 J**	**180 J**	750 U
Carbazole	NA	NA	NA	NA	750 U
Chrysene	**110 J**	559 U	917 U	**270 J**	**410 J**
Dibenzofuran	597 U	559 U	917 U	1,270 U	750 U
Di-n-butyl phthalate	597 U	559 U	917 U	**140 J**	**79 J**
Fluoranthene	**280 J**	**62 J**	**200 J**	**350 J**	**610 J**
Fluorene	597 U	559 U	917 U	1,270 U	750 U
Indeno(1,2,3-cd)pyrene	597 U	559 U	917 U	1,270 U	**200 J**
Naphthalene	597 U	559 U	917 U	1,270 U	750 U
Phenanthrene	**97 J**	559 U	917 U	1,270 U	**370 J**
Pyrene	**160 J**	**64 J**	**170 J**	**490 J**	**750 J**

See notes at end of table.

Table 5
Historical Sediment Analytical Data - VOCs and SVOCs
Koppers Pond, Horseheads, New York

1. *Operable Unit 3 RI data are from Philip Environmental (March 1996).*
2. *Only analytes detected in one or more samples are listed. Other VOC and SVOC analytes were not detected in any sediment sample above reporting limits.*
3. *For sampling locations, see Figure 3.*
4. *For clarity, all detections are shown in* ***bold-face type****.*
5. *"NA" indicates sample not analyzed for this compound.*
6. *All results are reported on a dry-weight basis.*
7. *Organic data qualifiers:*
   - *U - not detected at indicated detection limit*
   - *J - analyte detected, but concentration is an estimated value because the result is less than the quantitation limit or quality control criteria were not met.*
   - *B - constituent also detected in corresponding method blank*

Table 6
Historical Sediment Analytical Data - Pesticides and PCBs
Koppers Pond, Horseheads, New York

Parameter	Concentration by Sample Location and Sampling Date						
	SD-15 06/03/94	SD-16 06/02/94	SD-17 06/03/94	SD-18 06/02/94	SD-19 06/01/94	SD-20 06/01/94	SD-15B 06/06/95
Pesticides (ug/kg)							
4,4'-DDD	4.9 U	4.0 U	4.9 U	4.2 U	4.0 U	3.9 U	86.2 UJ
4,4'-DDE	4.9 U	4.0 U	4.9 U	**1.7 J**	4.0 U	3.9 U	86.2 UJ
4,4'-DDT	4.9 U	4.0 U	4.9 U	**0.62 JP**	4.0 U	3.9 U	86.2 UJ
Aldrin	2.5 U	2.1 U	2.5 U	2.2 U	2.1 U	2.0 U	43.1 UJ
Alpha-chlordane	2.5 U	2.1 U	2.5 U	**0.38 JP**	2.1 U	2.0 U	431 UJ
Delta-BHC	2.5 U	2.1 U	2.5 U	2.2 U	2.1 U	2.0 U	43.1 UJ
Dieldrin	4.9 U	4.0 U	4.9 U	4.2 U	4.0 U	3.9 U	86.2 UJ
Endosulfan II	4.9 U	4.0 U	4.9 U	**0.94 JP**	4.0 U	3.9 U	86.2 UJ
Endrin	4.9 U	4.0 U	4.9 U	**0.55 JP**	4.0 U	3.9 U	86.2 UJ
Endrin aldehyde	4.9 U	4.0 U	4.9 U	4.2 U	4.0 U	3.9 U	86.2 UJ
Endrin ketone	4.9 U	4.0 U	4.9 U	**1.5 J**	4.0 U	3.9 U	86.2 UJ
Gamma-chlordane	2.5 U	2.1 U	2.5 U	**1.1 JP**	2.1 U	2.0 U	431 UJ
Heptachlor	2.5 U	2.1 U	2.5 U	2.2 U	2.1 U	2.0 U	43.1 UJ
Heptachlor epoxide	2.5 U	2.1 U	2.5 U	**0.54 JP**	2.1 U	2.0 U	43.1 UJ
Lindane	2.5 U	2.1 U	2.5 U	2.2 U	2.1 U	2.0 U	43.1 UJ
Polychlorinated Biphenyls (ug/kg)							
Aroclor 1248	49 U	40 U	**150**	**5.6**	40 U	39 U	431 UJ
Aroclor 1254	**1,300**	40 U	**470**	**110**	40 U	39 U	**1,100 J**
Aroclor 1260	**310**	40 U	**170 P**	**51 P**	40 U	39 U	862 UJ
Total Organic Carbon (mg/kg)	NA	NA	NA	NA	NA	NA	NA

Table 6
Historical Sediment Analytical Data - Pesticides and PCBs
Koppers Pond, Horseheads, New York

Parameter	Concentration by Sample Location and Sampling Date						
	SD-16B 06/08/95	SD-17B 06/06/95	SD-18B 06/06/95	SD-19B 06/05/95	SD-20B 06/05/95	SD-01 08/17/98	SD-02 08/18/98
Pesticides (ug/kg)							
4,4'-DDD	**5.6 J**	54.6 UJ	91.3 UJ	**4.6 J**	**13 J**	4.3 UJ	**4.1 NJ**
4,4'-DDE	57.7 U	54.6 U	91.3 UJ	125 UJ	73.5 UJ	4.3 UJ	4.6 UJ
4,4'-DDT	57.7 U	54.6 U	91.3 UJ	125 UJ	73.5 UJ	4.3 UJ	**5.0 NJ**
Aldrin	28.9 U	27.3 U	45.6 UJ	62.5 UJ	36.8 UJ	2.2 UJ	2.4 UJ
Alpha-chlordane	289 U	273 U	456 UJ	625 UJ	368 UJ	2.2 UJ	2.4 UJ
Delta-BHC	28.9 U	27.3 U	45.6 UJ	62.5 UJ	36.8 UJ	**2.7 U**	2.4 UJ
Dieldrin	57.7 U	54.6 U	91.3 UJ	125 UJ	73.5 UJ	4.3 UJ	4.6 UJ
Endosulfan II	57.7 U	54.6 U	91.3 UJ	125 UJ	73.5 UJ	4.3 UJ	4.6 UJ
Endrin	57.7 UJ	54.6 UJ	91.3 UJ	125 UJ	73.5 UJ	4.3 UJ	4.6 UJ
Endrin aldehyde	57.7 UJ	54.6 UJ	91.3 UJ	125 UJ	73.5 UJ	4.3 UJ	**3.2 NJ**
Endrin ketone	57.7 U	54.6 U	91.3 UJ	125 UJ	73.5 UJ	4.3 UJ	4.6 UJ
Gamma-chlordane	289 U	273 U	456 UJ	625 UJ	368 UJ	2.2 UJ	**0.99 J**
Heptachlor	28.9 U	27.3 U	45.6 UJ	62.5 UJ	36.8 UJ	2.2 UJ	2.4 UJ
Heptachlor epoxide	28.9 U	27.3 U	45.6 UJ	62.5 UJ	36.8 UJ	2.2 UJ	2.4 UJ
Lindane	28.9 U	27.3 U	45.6 UJ	62.5 UJ	36.8 UJ	2.2 UJ	2.4 UJ
Polychlorinated Biphenyls (ug/kg)							
Aroclor 1248	289 U	273 U	456 UJ	625 UJ	368 UJ	43 UJ	46 UJ
Aroclor 1254	577 U	**170 J**	**1,200 J**	1,250 UJ	735 UJ	43 UJ	46 UJ
Aroclor 1260	577 U	546 U	913 UJ	1250 UJ	735 UJ	43 UJ	46 UJ
Total Organic Carbon (mg/kg)	NA	NA	NA	NA	NA	**48,010**	**27,100**

Table 6
Historical Sediment Analytical Data - Pesticides and PCBs
Koppers Pond, Horseheads, New York

Parameter	Concentration by Sample Location and Sampling Date						
	SD-03 08/18/98	SD-04 08/18/98	SD-05 08/18/98	SD-06 08/19/98	SD-07 08/19/98	SD-08 08/20/98	SD-08 (0-3) 08/20/98
Pesticides (ug/kg)							
4,4'-DDD	17 UJ	R	15 UJ	140 UJ	97 UJ	130 UJ	160 UJ
4,4'-DDE	17 UJ	5.5 UJ	15 UJ	140 UJ	97 UJ	130 UJ	160 UJ
4,4'-DDT	R	**22 J**	**38 J**	140 UJ	97 UJ	**170 J**	**270 J**
Aldrin	8.9 UJ	2.8 UJ	7.8 UJ	71 UJ	50 UJ	65 UJ	85 UJ
Alpha-chlordane	8.9 UJ	2.8 UJ	7.8 UJ	71 UJ	50 UJ	**54 J**	**160 J**
Delta-BHC	**9.5 J**	2.8 UJ	7.8 UJ	71 UJ	50 UJ	65 UJ	85 UJ
Dieldrin	17 UJ	5.5 UJ	15 UJ	140 UJ	97 UJ	130 UJ	160 UJ
Endosulfan II	17 UJ	5.5 UJ	**9 J**	140 UJ	97 UJ	130 UJ	160 UJ
Endrin	17 UJ	5.5 UJ	**6.5 J**	140 UJ	97 UJ	**90 J**	160 UJ
Endrin aldehyde	17 UJ	**5.1 NJ**	15 UJ	140 UJ	97 UJ	130 UJ	**40 NJ**
Endrin ketone	17 UJ	5.5 UJ	15 UJ	140 UJ	97 UJ	130 UJ	160 UJ
Gamma-chlordane	**11 J**	**3.2 J**	7.8 UJ	71 UJ	50 UJ	**100 J**	**240 J**
Heptachlor	**7.8 J**	2.8 UJ	7.8 UJ	71 UJ	50 UJ	65 UJ	85 UJ
Heptachlor epoxide	8.9 UJ	**1.9 J**	7.8 UJ	71 UJ	50 UJ	65 UJ	85 UJ
Lindane	8.9 UJ	2.8 UJ	7.8 UJ	71 UJ	50 UJ	65 UJ	85 UJ
Polychlorinated Biphenyls (ug/kg)							
Aroclor 1248	170 UJ	55 UJ	150 UJ	1,400 UJ	970 UJ	1,300 UJ	1,600 UJ
Aroclor 1254	170 UJ	55 UJ	150 UJ	**410 J**	**220 J**	**1,500 J**	**2,900 J**
Aroclor 1260	170 UJ	55 UJ	150 UJ	1,400 UJ	970 UJ	1,300 UJ	1,600 UJ
Total Organic Carbon (mg/kg)	**101,800**	**40,370**	**101,300**	**106,500**	**96,870**	**101,800**	NA

Table 6
Historical Sediment Analytical Data - Pesticides and PCBs
Koppers Pond, Horseheads, New York

Parameter	Concentration by Sample Location and Sampling Date						
	SD-08 (3-9) 08/20/98	SD-08 (9-12) 08/20/98	SD-08 (12-17) 08/20/98	SD-09 08/20/98	SD-09 (0-6) 08/20/98	SD-09 (6-12) 08/20/98	SD-10 08/19/98
Pesticides (ug/kg)							
4,4'-DDD	100 UJ	100 UJ	69 UJ	120 UJ	110 UJ	72 UJ	180 UJ
4,4'-DDE	100 UJ	**110 NJ**	69 UJ	120 UJ	110 UJ	72 UJ	180 UJ
4,4'-DDT	**190 J**	**570 J**	**45 J**	**120 J**	110 UJ	**100 J**	180 UJ
Aldrin	53 UJ	52 UJ	35 UJ	61 UJ	**28 J**	37 UJ	94 UJ
Alpha-chlordane	**53 J**	**170 J**	35 UJ	**40 J**	59 UJ	**33 J**	94 UJ
Delta-BHC	53 UJ	52 UJ	35 UJ	61 UJ	59 UJ	37 UJ	94 UJ
Dieldrin	100 UJ	**38 J**	69 UJ	120 UJ	110 UJ	72 UJ	180 UJ
Endosulfan II	100 UJ	**250 J**	69 UJ	120 UJ	110 UJ	72 UJ	180 UJ
Endrin	**89 J**	**260 J**	**24 J**	**71 J**	**90 J**	**57 J**	180 UJ
Endrin aldehyde	**68 NJ**	100 UJ	69 UJ	120 UJ	110 UJ	**40 NJ**	180 UJ
Endrin ketone	**92 NJ**	100 UJ	69 UJ	120 UJ	110 UJ	72 UJ	180 UJ
Gamma-chlordane	**120 J**	**250 J**	**28 J**	**80 J**	59 UJ	**76 J**	94 UJ
Heptachlor	53 UJ	52 UJ	35 UJ	61 UJ	**48 J**	37 UJ	94 UJ
Heptachlor epoxide	53 UJ	52 UJ	35 UJ	61 UJ	59 UJ	37 UJ	94 UJ
Lindane	53 UJ	52 UJ	35 UJ	61 UJ	**39 J**	37 UJ	94 UJ
Polychlorinated Biphenyls (ug/kg)							
Aroclor 1248	1,000 UJ	1,000 UJ	690 UJ	1,200 UJ	1,100 UJ	720 UJ	1,800 UJ
Aroclor 1254	**1,500 J**	**4,100 J**	**400 J**	**1,100 J**	**320 J**	**320 J**	**730 J**
Aroclor 1260	1,000 UJ	1,000 UJ	690 UJ	1,200 UJ	1,100 UJ	720 UJ	1,800 UJ
Total Organic Carbon (mg/kg)	NA	NA	NA	**103,200**	NA	NA	**205,800**

Table 6
Historical Sediment Analytical Data - Pesticides and PCBs
Koppers Pond, Horseheads, New York

Parameter	Concentration by Sample Location and Sampling Date							
	SD-11 08/20/98	SD-12 08/19/98	SD-12 (0-6) 08/19/98	SD-12 (6-12) 08/19/98	SD-12 (12-18) 08/19/98	SD-12 (18-21) 08/19/98	SD-13 08/19/98	SD-20 8/201998
Pesticides (ug/kg)								
4,4'-DDD	110 UJ	110 UJ	120 UJ	100 UJ	89 UJ	100 UJ	120 UJ	110 UJ
4,4'-DDE	110 UJ	110 UJ	120 UJ	100 UJ	89 UJ	100 UJ	120 UJ	110 UJ
4,4'-DDT	**110 J**	**140 J**	120 UJ	**140 J**	89 UJ	100 UJ	**140 J**	**480 J**
Aldrin	57 UJ	57 UJ	**33 J**	52 UJ	46 UJ	52 UJ	63 UJ	57 UJ
Alpha-chlordane	**36 J**	**42 J**	61 UJ	**38 J**	46 UJ	52 UJ	63 UJ	**180 J**
Delta-BHC	57 UJ	57 UJ	61 UJ	52 UJ	46 UJ	52 UJ	63 UJ	57 UJ
Dieldrin	110 UJ	110 UJ	120 UJ	100 UJ	89 UJ	100 UJ	120 UJ	110 UJ
Endosulfan II	110 UJ	110 UJ	120 UJ	100 UJ	89 UJ	100 UJ	120 UJ	110 UJ
Endrin	**62 J**	**72 J**	**100 J**	**70 J**	89 UJ	100 UJ	120 UJ	**280 J**
Endrin aldehyde	110 UJ	**47 NJ**	120 UJ	100 UJ	89 UJ	100 UJ	120 UJ	110 UJ
Endrin ketone	110 UJ	110 UJ	120 UJ	100 UJ	89 UJ	100 UJ	120 UJ	**62 NJ**
Gamma-chlordane	**83 J**	**98 J**	61 UJ	**87 J**	46 UJ	52 UJ	**87 NJ**	**280 J**
Heptachlor	57 UJ	57 UJ	**56 J**	52 UJ	46 UJ	52 UJ	63 UJ	57 UJ
Heptachlor epoxide	57 UJ	57 UJ	61 UJ	52 UJ	46 UJ	52 UJ	63 UJ	57 UJ
Lindane	57 UJ	57 UJ	**45 J**	52 UJ	46 UJ	52 UJ	63 UJ	57 UJ
Polychlorinated Biphenyls (ug/kg)								
Aroclor 1248	1,100 UJ	1,100 UJ	1,200 UJ	1,000 UJ	890 UJ	1,000 UJ	1,200 UJ	1,000 UJ
Aroclor 1254	**1,100 J**	**1,200 J**	1,200 UJ	**1,100 J**	**290 J**	1,000 UJ	**1,100 J**	**4,500 J**
Aroclor 1260	1,100 UJ	1,100 UJ	1,200 UJ	1,000 UJ	890 UJ	1,000 UJ	1,200 UJ	1,000 UJ
Total Organic Carbon (mg/kg)	**132,200**	**104,400**	NA	NA	NA	NA	**135,700**	**151,450**

Table 6
Historical Sediment Analytical Data - Pesticides and PCBs
Koppers Pond, Horseheads, New York

1. *Operable Unit 3 RI data are from Philip Environmental (March 1996).*
   1998 data are from CDM (February 1999).
2. *Only analytes detected in one or more samples are listed. Other pesticide and PCB analytes were not detected in any sediment sample above reporting limits.*
3. *For sampling locations, see Figure 3.*
4. *For clarity, all detections are shown in* ***bold-face type*** *.*
5. *All results are reported on a dry-weight basis.*
6. *Organic data qualifiers:*
   U - not detected at indicated reporting limit.
   J - analyte detected, but concentration is an estimated value because the result is less than the quantitation limit or quality control criteria were not met.
   P - percent difference between results from both columns was greater than 25 percent.
   N - compound is presumed to be present based on analytical evidence.

Table 7
Fish Tissue Analytical Data - June 1995 Sampling
Koppers Pond, Horseheads, New York

Parameter	Concentration in Prepared Tissue Sample						
	CC-01	CC-02	CC-03	CC-04	CC-05	CC-06	CC-COMP
Volatile Organic Compounds (ug/kg)							
Acetone	**194 J**	**19.8 J**	50 UJ	50 UJ	50 UJ	**73.9 J**	95 UJ
Carbon disulfide	**98.5 J**	**183 J**	**133**	**218 J**	**66.8 J**	**126 J**	**109 J**
Toluene	25 U	**5.82 J**	**10.5 J**	25 UJ	25 UJ	**11.50 J**	25 UJ
Tetrachloroethylene	25 U	25 U	25 UJ	25 UJ	25 UJ	25 UJ	25 UJ
Polychlorinated Biphenyls (ug/kg)							
Aroclor 1254	**191**	201 U	**93.1 J**	**64.0 J**	**96.5 J**	335 U	**234**
Metals (mg/kg)							
Aluminum	**1.13 B**	NA	NA	NA	NA	NA	NA
Arsenic	0.018 UB	NA	NA	NA	NA	NA	NA
Barium	0.23 U	NA	NA	NA	NA	NA	NA
Calcium	**221 B**	NA	NA	NA	NA	NA	NA
Chromium	**0.468 B**	NA	NA	NA	NA	NA	NA
Copper	**0.425 B**	NA	NA	NA	NA	NA	NA
Iron	**4.64 B**	NA	NA	NA	NA	NA	NA
Lead	**0.849 B**	NA	NA	NA	NA	NA	NA
Magnesium	**136 B**	NA	NA	NA	NA	NA	NA
Manganese	**0.119 B**	NA	NA	NA	NA	NA	NA
Mercury	0.040 U	NA	NA	NA	NA	NA	NA
Nickel	**3.11**	NA	NA	NA	NA	NA	NA
Potassium	**2,140**	NA	NA	NA	NA	NA	NA
Sodium	**305**	NA	NA	NA	NA	NA	NA
Zinc	**4.78 B**	NA	NA	NA	NA	NA	NA

See notes at end of table.

Table 7
Fish Tissue Analytical Data - June 1995 Sampling
Koppers Pond, Horseheads, New York

Parameter	Concentration in Prepared Tissue Sample					
	WS-01	WS-02	WS-03	WS-04	WS-05	WS-06
Volatile Organic Compounds (ug/kg)						
Acetone	192 J	203 J	58.0 J	474 J	95 UJ	31.3 J
Carbon disulfide	27.5	92.5	23.9 J	589	77.8 J	223
Toluene	25 U	25 U	25 U	25 UJ	25 UJ	8.09 UJ
Tetrachloroethylene	8.06 J	25 U	25 U	25 UJ	25 UJ	25 U
Polychlorinated Biphenyls (ug/kg)						
Aroclor 1254	537 J	298 J	374	392	105	187 J
Metals (mg/kg)						
Aluminum	60.4 B	1.21 B	1.39 B	77.3 B	NA	NA
Arsenic	0.042 B	0.018 U	0.038 B	0.098 B	NA	NA
Barium	0.45 U	0.228 U	0.456 B	0.51 U	NA	NA
Calcium	506 B	195 B	222 B	460 B	NA	NA
Chromium	0.997 B	0.869 B	0.442 B	0.946 B	NA	NA
Copper	2.28 B	1.25 B	0.818 B	3.69 B	NA	NA
Iron	9.53 B	6.33 B	4.34 B	5.76 B	NA	NA
Lead	0.664 B	0.161 B	0.427 B	0.607 B	NA	NA
Magnesium	270 B	132 B	144 B	217 B	NA	NA
Manganese	0.320 B	0.116 B	0.104 B	0.694 B	NA	NA
Mercury	0.040 U	0.040 U	0.058 B	0.040 U	NA	NA
Nickel	1.50 B	0.707 B	1.44 B	0.68 B	NA	NA
Potassium	4,510 B	2,160 B	2,350 B	3,610 B	NA	NA
Sodium	750 B	369 B	298 B	628 B	NA	NA
Zinc	14.5 B	6.67 B	8.12 B	14.8 B	NA	NA

See notes at end of table.

Table 7
Fish Tissue Analytical Data - June 1995 Sampling
Koppers Pond, Horseheads, New York

1. *Data from Philip Environmental (March 1996). See Appendix H, Table 4 of that report for inventory of fish samples, including sample numbers, species, weight, length, and sex.*
2. *"CC" refers to Common Carp samples. "WS" refers to White Sucker samples. "CC-COMP" is a laboratory formulated composite sample from samples C-07, CC-08, and CC-09.*
3. *Only analytes detected in one or more samples are listed. Other analytes (e.g., SVOCs) were not detected in any sample above reporting limits.*
4. *For clarity, all detections are shown in* ***bold-face type****.*
5. *"NA" indicates sample not analyzed for this constituent.*
6. *Organic data qualifiers:*
   - *U - not detected at indicated detection limit.*
   - *J - analyte detected, but concentration is an estimated value because the result is less than the quantitation limit or quality control criteria were not met.*
7. *Inorganic data qualifiers:*
   - *U - not detected at indicated detection limit.*
   - *B - detected concentration below quantitation limit but above instrument detection limit.*

Table 8
Fish Tissue Analytical Data - July 2003 Sampling
Koppers Pond, Horseheads, New York

Parameter	Concentration by Species and Sample Number						
	Common Carp					White Sucker	
	CC-1-LS	CC-2-LS	CC-3-LS	CC-4-RS	CC-5-RS	WS-1-LS	WS-2-LS
Polychlorinated Biphenyls (ug/kg)							
Aroclor 1248	150	200 U	120 J	250 U	100 U	47 J	50 U
Aroclor 1254	530	1,000	1,300	2,000	570	590	310
Aroclor 1260	170	160 J	230 J	400	76 J	80 J	34 J
Total PCBs	850	1,160	1,650	2,400	646	717	344
Metals (mg/kg)							
Aluminum	4.5 B	4.4 B	3.1 B	4.8 B	3.1 B	8.0 B	4.2 B
Antimony	0.16 B	1.3 U	1.3 U	0.32 B	1.3 U	1.4 U	1.5 U
Arsenic	1.5 U	1.3 U	1.3 U	1.5 U	1.3 U	1.4 U	1.5 U
Barium	2.0	2.1	2.9	0.99	1.9	2.1	1.1
Beryllium	0.30 U	0.27 U	0.26 U	0.29 U	0.26 U	0.29 U	0.30 U
Cadmium	0.15 B	0.093 B	0.056 B	0.095 B	0.033 B	0.27 B	0.30 U
Calcium	4,200	4,600	6,800	3,900	4,700	6,800	4,800
Chromium	0.29 B	0.21 B	0.24 B	0.24 B	0.16 B	0.56	0.16 B
Cobalt	0.50 U	0.45 U	0.43 U	0.49 U	0.44 U	0.48 U	0.50 U
Copper	1.1	0.93	1.1	1.3	0.71 B	1.4	0.45 B
Iron	15 B	7.9 B	7.7 B	12 B	3.9 B	13 B	2.3 B
Lead	0.68 B	0.90	1.60	0.59 B	0.52 B	1.40	0.35 B
Magnesium	260	290	310	250	310	370	310
Manganese	2.0	0.40	0.52	1.6	0.47	0.24	0.40
Mercury	0.035	0.0087 B	0.0044 B	0.10 B	0.0044 B	0.0098 B	0.0086 B
Nickel	0.99 U	0.89 U	0.87 U	0.98 U	0.88 U	0.95 U	0.99 U
Potassium	2,900	2,900	2,700	1,800	3,100	3,500	3,200
Selenium	0.37 B	0.67 B	0.56 B	0.69 B	0.34 B	0.43 B	0.44 B
Silver	0.49 U	0.44 U	0.43 U	0.48 U	0.45 U	0.093 B	0.50 U
Sodium	730	710	770	990	750	690	690
Zinc	15	30	23	19	23	15	9.6

See notes at end of table.

Table 8
Fish Tissue Analytical Data - July 2003 Sampling
Koppers Pond, Horseheads, New York

Parameter	Concentration by Species and Sample Number						
	White Sucker			Largemouth Bass		Pumpkinseed	
	WS-3-LS	WS-4-RS	WS-5-RS	LB-1-RS	LB-2-RS	PS-1	PS-2
Polychlorinated Biphenyls (ug/kg)							
Aroclor 1248	100 U	210	200 U	50 U	29 J	63	86
Aroclor 1254	570	160	1,300	420	180	420	410
Aroclor 1260	74 J	38 J	200 J	86	58	77 J	99
Total PCBs	644	408	1,500	506	267	560	595
Metals (mg/kg)							
Aluminum	4.4 B	19 U	3.4 B	18 U	17 U	15 B	18 B
Antimony	1.3 U	1.4 U	0.45 B	1.4 U	1.3 U	0.27 B	0.28 B
Arsenic	1.3 U	1.4 U	1.4 U	1.4 U	1.3 U	1.5 U	1.5 U
Barium	0.6	1.3	1.5	0.43	0.71	2.3	3.1
Beryllium	0.26 U	0.28 U	0.28 U	0.27 U	0.26 U	0.30 U	0.30 U
Cadmium	0.053 B	0.28 U	0.030 B	0.27 U	0.26 U	0.14 B	0.54
Calcium	1,300	3,800	7,100	5,800	7,300	12,000	16,000
Chromium	0.17 B	0.16 B	0.3	0.13 B	0.23 B	0.58	1.0
Cobalt	0.43 U	0.47 U	0.46 U	0.45 U	0.43 U	0.065 B	0.50 U
Copper	0.67 B	0.57 B	1.1	0.51 B	0.67 B	2.2	1.5
Iron	4.7 B	4.1 B	8.8 B	3.3 B	3.0 B	21	29
Lead	0.56 B	0.32 B	0.55 B	0.19 B	0.17 B	1.1	2.1
Magnesium	280	310	360	300	320	390	470
Manganese	0.15 B	0.25	0.24	0.11 B	0.18	1.1	1.1
Mercury	0.0041 B	0.012	0.0045 B	0.056	0.091	0.011	0.0095 B
Nickel	0.85 U	0.93 U	0.24 B	0.91 U	0.85 U	0.23 B	0.38 B
Potassium	3,400	3,200	3,300	3,100	2,900	2,700	2,700
Selenium	0.59 B	0.43 B	0.55 B	0.47 B	1.3 U	0.64 B	0.80 B
Silver	0.50 U	0.47 U	0.47 U	0.49 U	0.43 U	0.50 U	0.064 B
Sodium	560	640	790	790	800	940	1,000
Zinc	8.4	10	11	9.6	8.0	27	33

See notes at end of table.

Table 8
Fish Tissue Analytical Data - July 2003 Sampling
Koppers Pond, Horseheads, New York

Parameter	Concentration by Species and Sample Number					
	Pumpkinseed				Black Crappie	
	PS-3	PS-4	PS-5	PS-6	BC-1-RS	BC-2-RS
Polychlorinated Biphenyls (ug/kg)						
Aroclor 1248	140 U	1,100	50 U	100 U	50 U	50 U
Aroclor 1254	950	200 U	200	670	220	490
Aroclor 1260	330	82 J	140	200	130	120
Total PCBs	1,280	1,182	340	870	350	610
Metals (mg/kg)						
Aluminum	12 B	14 B	70	7.2 B	3.2 B	3.3 B
Antimony	0.26 B	0.17 B	0.28 B	0.35 B	0.33 B	1.2 U
Arsenic	1.3 U	1.3 U	0.33 B	1.4 U	1.3 U	1.2 U
Barium	3.1	2.7	3.8	1.7	2.4	3.8
Beryllium	0.26 U	0.26 U	0.25 U	0.29 U	0.27 U	0.25 U
Cadmium	0.084 B	0.11 B	0.42	0.090 B	0.27 U	0.088 B
Calcium	16,000	15,000	16,000	18,000	18,000	20,000
Chromium	0.60	0.69	0.74	0.50	0.35	0.46
Cobalt	0.44 U	0.44 U	0.13 B	0.48 U	0.44 U	0.42 U
Copper	0.76 B	0.98	1.3	0.52 B	0.48 B	0.46 B
Iron	26	24	220	18	7.8 B	9.5 B
Lead	1.1	1.2	1.6	1.0	0.63 B	0.64 B
Magnesium	440	440	450	440	490	510
Manganese	1.0	1.6	5.8	0.6	1.9	1.8
Mercury	0.0087 B	0.013	0.019	0.038	0.047	0.022
Nickel	0.88 U	0.31 B	0.35 B	0.95 U	0.24 B	0.83 U
Potassium	2,500	2,600	2,300	2,400	2,500	2,300
Selenium	0.58 B	0.61 B	0.84 B	0.39 B	0.56 B	0.80 B
Silver	0.50 U	0.43 U	0.097 B	0.45 U	0.47 U	0.45 U
Sodium	1,400	1,100	1,400	1,300	1,100	970
Zinc	27	27	28	27	24	29

See notes at end of table.

Table 8
Fish Tissue Analytical Data - July 2003 Sampling
Koppers Pond, Horseheads, New York

Parameter	Concentration by Species and Sample Number			
	Black Crappie	Green Sunfish		
	BC-3-RS	GS-1-RS	GS-2-RS	GS-3-RS
Polychlorinated Biphenyls (ug/kg)				
Aroclor 1248	**52**	50 U	50 U	**56**
Aroclor 1254	**480**	**360**	**480**	**240**
Aroclor 1260	**110**	**94**	**130**	**110**
Total PCBs	**642**	**454**	**610**	**406**
Metals (mg/kg)				
Aluminum	17 U	20 U	**23**	**14 B**
Antimony	1.2 U	**0.37 B**	1.3 U	1.3 U
Arsenic	1.2 U	1.5 U	1.3 U	1.3 U
Barium	**3.3**	**4.0**	**1.7**	**2.0**
Beryllium	0.25 U	0.30 U	0.27 U	0.27 U
Cadmium	**0.030 B**	**0.051 B**	**0.30**	**0.12 B**
Calcium	**15,000**	**18,000**	**12,000**	**13,000**
Chromium	**0.35**	**0.42**	**0.40**	**0.58**
Cobalt	0.42 U	0.50 U	**0.11 B**	**0.14 B**
Copper	**0.56 B**	**0.56 B**	**0.78 B**	**0.82 B**
Iron	**9.2 B**	**7.3 B**	**52**	**35**
Lead	**0.34 B**	**0.63 B**	**0.91**	**0.99**
Magnesium	**450**	**490**	**400**	**400**
Manganese	**1.6**	**1.9**	**1.6**	**1.1**
Mercury	**0.012**	**0.015**	**0.052**	**0.037**
Nickel	0.83 U	0.99 U	**0.20 B**	**0.30 B**
Potassium	**2,500**	**2,400**	**2,500**	**2,400**
Selenium	**0.62 B**	**0.52 B**	**0.58 B**	**0.96 B**
Silver	0.46 U	0.44 U	0.42 U	0.43 U
Sodium	**940**	**990**	**1,100**	**1,200**
Zinc	**28**	**32**	**32**	**22**

See notes at end of table.

Table 8
Fish Tissue Analytical Data - July 2003 Sampling
Koppers Pond, Horseheads, New York

1. *Data from CEC (July 2003). See Table 1 of that report for inventory of fish samples, including sample numbers, species, weight, and length.*
2. *Only analytes detected in one or more samples are listed. Other analytes were not detected in any sample above reporting limits.*
3. *For clarity, all detections are shown in* ***bold-face type***.
4. *Organic data qualifiers:*
   U - not detected at indicated detection limit.
   J - analyte detected, but concentration is an estimated value because the result is less than the quantitation limit or quality control criteria were not met.
5. *Inorganic data qualifiers:*
   U - not detected at indicated detection limit.
   B - detected concentration below quantitation limit but above instrument detection limit.

Table 9
New York State Ambient Water Quality Criteria for Class C Surface Waters
Koppers Pond, Horseheads, New York

Substance	CAS No.	Standard (µg/L)	Type	Notes
Aldrin and Dieldrin	309-00-2 60-57-1	0.001	H(FC)	Standard applies to the sum of these substances.
Aluminum, ionic	NA	100	A(C)	
Ammonia	7664-41-7	*	A(C)	* Un-ionized ammonia as NH_3; tables below (in notes) provide the standard in ug/L at varying pH and temperature. Linear interpolation between the listed pH values and temperatures is applicable.
Arsenic	NA	150 340	A(C) A(A)	Standards apply to the dissolved form.
Benzene	71-43-2	10	H(FC)	
Beryllium	NA	*	A(C)	* 11 ug/L, when hardness is less than or equal to 75 ppm; 1,100 ug/L when hardness is greater than 75 ppm. Standard applies to acid-soluble form.
Bis(2-ethylhexyl)phthalate	117-81-7	0.6	A(C)	
Cadmium	NA	* **	A(C) A(A)	* (0.85) exp (0.7852 [ln (ppm hardness)] - 2.715) ** (0.85) exp (1.128 [ln (ppm hardness)] - 3.6867) Standards apply to the dissolved form.
Chlorobenzene	108-90-7	400 5	H(FC) A(C)	
Chromium	NA	* **	A(C) A(A)	* (0.86) exp (0.819 [ln (ppm hardness)] + 0.6848) ** (0.316) exp (0.819 [ln (ppm hardness)] + 3.7256) Standards apply to dissolved form and do not include hexavalent chromium.
Chromium (hexavalent)	NA	11 16	A(C) A(A)	Standard applies to the acid-soluble form.

Table 9
New York State Ambient Water Quality Criteria for Class C Surface Waters
Koppers Pond, Horseheads, New York

Substance	CAS No.	Standard (µg/L)	Type	Notes
Cobalt	NA	5*	A(C)	
Coliforms, Fecal	NA	*	G	* The monthly geometric mean, from a minimum of five examinations, shall not exceed 200.
Coliforms, Total	NA	*	G	* The monthly median value and more than 20 percent of the samples, from a minimum of five examinations, shall not exceed 2,400 and 5,000, respectively.
Copper	NA	* **	A(C) A(A)	* (0.96) exp (0.8545 [ln (ppm hardness)] - 1.702) ** (0.96) exp (0.9422 [ln (ppm hardness)] - 1.7) Standard applies to the dissolved form.
Cyanide	NA	9,000 5.2* 22*	H(FC) A(C) A(A)	* As free cyanide: the sum of HCN and CN- expressed as CN.
p,p'-DDD	72-54-8	8×10^{-5} *	H(FC) W	* See standard for p,p'-DDT.
p,p'-DDE	72-55-9	7×10^{-6} *	H(FC) W	* See standard for p,p'-DDT.
p,p'-DDT	50-29-3	1×10^{-5} 1.1×10^{-5}*	H(FC) W	* Standard applies to the sum of p,p'-DDD, p,p'-DDE and p,p'-DDT.
Dichlorobenzenes	95-50-1 541-73-1 106-46-7	5**	A(C)	** Standard applies to the sum of 1,2-, 1,3- and 1,4-dichlorobenzene.
2,4-Dichlorophenol	120-83-2	*	E	*Refer to standards for "Phenols, total chlorinated"
Dieldrin	60-57-1	6×10^{-7} 0.056 0.24	H(FC) A(C) A(A)	

Table 9
New York State Ambient Water Quality Criteria for Class C Surface Waters
Koppers Pond, Horseheads, New York

Substance	CAS No.	Standard (µg/L)	Type	Notes
2,4-Dimethylphenol	105-67-9	1,000 **	H(FC) E	** Refer to standard for "Phenols, total unchlorinated."
2,4-Dinitrophenol	51-28-5	400 **	H(FC) E	** Refer to standard for "Phenols, total unchlorinated."
Endosulfan	115-29-7	0.009	A(C)	
Endrin	72-20-8	0.002 0.036 0.086	H(FC) A(C) A(A)	
Fluoride	NA	*	A(C)	* (0.02) exp(0.907 [ln (ppm hardness)] + 7.394)
Heptachlor	76-44-8	2×10^{-4}	H(FC)	
Heptachlor epoxide	1024-57-3	3×10^{-4}	H(FC)	
Hexachlorobenzene	118-74-1	3×10^{-5}	H(FC)	
Hexachlorobutadiene	87-68-3	0.01 1.0*	H(FC) A(C)	
α-Hexachlorocyclohexane	319-84-6	0.002	H(FC)	
β-Hexachlorocyclohexane	319-85-7	0.007	H(FC)	
δ-Hexachlorocyclohexane	319-86-8	0.008	H(FC)	
γ-Hexachlorocyclohexane	58-89-9	0.008 0.95	H(FC) A(A)	
Hexachlorocyclopentadiene	77-47-4	0.45**	A(C)	
Hexachloroethane	67-72-1	0.6	H(FC)	
Iron	NA	300**	A(C)	

Table 9
New York State Ambient Water Quality Criteria for Class C Surface Waters
Koppers Pond, Horseheads, New York

Substance	CAS No.	Standard (µg/L)	Type	Notes
Lead	NA	*	A(C)	* {1.46203 - [ln (hardness) (0.145712)]} exp (1.273 [ln (hardness)] - 4.297)
		**	A(A)	** {1.46203 - [ln (hardness) (0.145712)]} exp (1.273 [ln (hardness)] - 1.052)
				Standards apply to dissolved form.
Mercury	NA	7 x 10-4*	H(FC)	* Standards apply to dissolved form.
		0.77*	A(C)	
		1.4*	A(A)	
		0.0026*	W	
Methylene chloride	75-09-2	200	H(FC)	
Nickel	NA	*	A(C)	* (0.997) exp (0.846 [ln (hardness)] + 0.0584)
		**	A(A)	** (0.998) exp (0.846 [ln (hardness)] + 2.255)
				Standards apply to dissolved form.
Nitrite (expressed as N)	NA	**	A(C)	** Standard is 100 ug/L for warm water fishery waters and 20 ug/L for cold water fishery waters.
Oxygen, dissolved	NA	*	G	* For cold waters suitable for trout spawning, the DO concentration shall not be less than 7.0 mg/L from other than natural conditions. For trout waters, the minimum daily average shall not be less than 6.0 mg/L, and at no time shall the concentration be less than 5.0 mg/L. For nontrout waters, the minimum daily average shall not be less than 5.0 mg/L, and at no time shall the DO concentration be less than 4.0 mg/L.
Pentachlorophenol	87-86-5	*	A(C)	* exp [1.005 (pH) - 5.134]
		**	A(A)	** exp [1.005 (pH) - 4.869]
		****	E	**** Refer to standards for "Phenols, total chlorinated."

Table 9
New York State Ambient Water Quality Criteria for Class C Surface Waters
Koppers Pond, Horseheads, New York

Substance	CAS No.	Standard (µg/L)	Type	Notes
Phenol	108-95-2	**	E	** Refer to standards for "Phenols, total unchlorinated."
pH	NA	*	G	* 6.5 < pH < 8.5
Polychlorinated biphenyls		1×10^{-6}* 1.2×10^{-4}*	H(FC) W	** Applies to the sum of these substances.
Selenium		4.6*	A(C)	* Standard applies to dissolved form.
Silver		0.1*	A(C)	* Standard applies to ionic silver.
Solids	NA	*	G	* Shall be kept as low as practicable to maintain the best usage of waters but in no case shall it exceed 500 mg/L.
Thallium	NA	8*	A(C)	Standard applies to acid-soluble form.
Toluene	(108-88-3)	6000	H(FC)	
Toxaphene	(8001-35-2)	6×10^{-6} 0.005	H(FC) A(C)	
Trichlorobenzenes	87-61-6 120-82-1 108-70-3 12002-48-1	5**	A(C)	** Applies to the sum of 1,2,3-, 1,2,4- and 1,3,5-trichlorobenzene.
Trichloroethylene	(79-01-6)	40	H(FC)	
Vanadium	NA	14	A(C)	Standard applies to acid-soluble form.
Zinc	NA	* **	A(C) A(A)	* exp(0.85 [ln(ppm hardness)] + 0.50) ** 0.978 exp(0.8473 [ln(ppm hardness)] + 0.884) Standards apply to dissolved form.

Table 9
New York State Ambient Water Quality Criteria for Class C Surface Waters
Koppers Pond, Horseheads, New York

Notes:

1. Criteria from 10 NYCRR Part 703.
2. Substances listed are those included in TCL/TAL list plus general chemistry parameters identified for surface water analysis.
3. Ammonia criteria (un-ionized ammonia as NH_3) are as follows (ug/L) at varying pH and temperature. Linear interpolation between the listed pH values and temperatures is applicable.

pH	0°C	5°C	10°C	15°C	20-30°C
6.5	0.7	0.9	1.3	1.9	2.6
6.75	1.2	1.7	2.3	3.3	4.7
7	2.1	2.9	4.2	5.9	8.3
7.25	3.7	5.2	7.4	11	15
7.5	6.6	9.3	13	19	26
7.75	11	15	22	31	43
8.0-9.0	13	18	25	35	50

Table 10
Candidate Receptors for SBERA
Koppers Pond, Horseheads, New York

Group	Feeding Guild	Receptors
Aquatic Organisms		Amphibians - green frog (*Rana clamitans melanota*)
		Reptiles - painted turtle (*Chrysemys picta marginata*)
		Fish - forage fish, bottom- and water column-dwelling fish
Avian Receptors	Avian piscivore	Belted kingfisher (*Ceryle alcyon*)
Mammalian Receptors	Mammalian omnivoire	Raccoon (*Procyon lotor*)
	Mammalian piscivore	Mink (*Mustela vison*)

TABLE 11

SUMMARY OF SAMPLING PROGRAM

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS[a]	SAMPLES	FIELD DUPLICATES	MS/MSD SAMPLES	EQUIPMENT RINSATE BLANKS[b]	TRIP BLANKS[c]
Surface Water and Seeps	Oxidation/ Reduction Potential, pH, Temp., Specific Conductance, Dissolved Oxygen	Full TCL/TAL Plus ammonia, fluoride, hardness, nitrites, and total suspended solids	13[d]	1[d]	1[d]	1[d]	1[d]
Sediment[e]	pH, Oxidation/ Reduction Potential	Full TCL/TAL, and total organic carbon	17-44	2	2	2	2
Fish Tissue	--	TCL PCBs/Pesticides, TAL, lipid content[f]	12-24	--	1-2	--	--
Pipe Floc	--	Full TCL/TAL	1	--	--	--	--
Barrier Well Treated Discharge/ Cutler-Hammer Discharge/ Chemung Street Outfall	Oxidation/ Reduction Potential, pH, Temp., Specific Conductance, Dissolved Oxygen	Full TCL/TAL Plus ammonia, fluoride, hardness, nitrites, and total suspended solids	1 (each potential source)	--	--	--	--

(a) Parameters include: Full TCL/TAL includes VOCs, SVOCs, pesticides/PCBs, and TAL inorganics. TAL inorganics analyses of aqueous samples will include both the dissolved and total fractions.

(b) Equipment rinsate blanks will not be collected if disposable sampling tools are used.

(c) One trip blank will be shipped with each container submitted to the laboratory for VOC analyses. The total number of trip blanks in the table is an estimate.

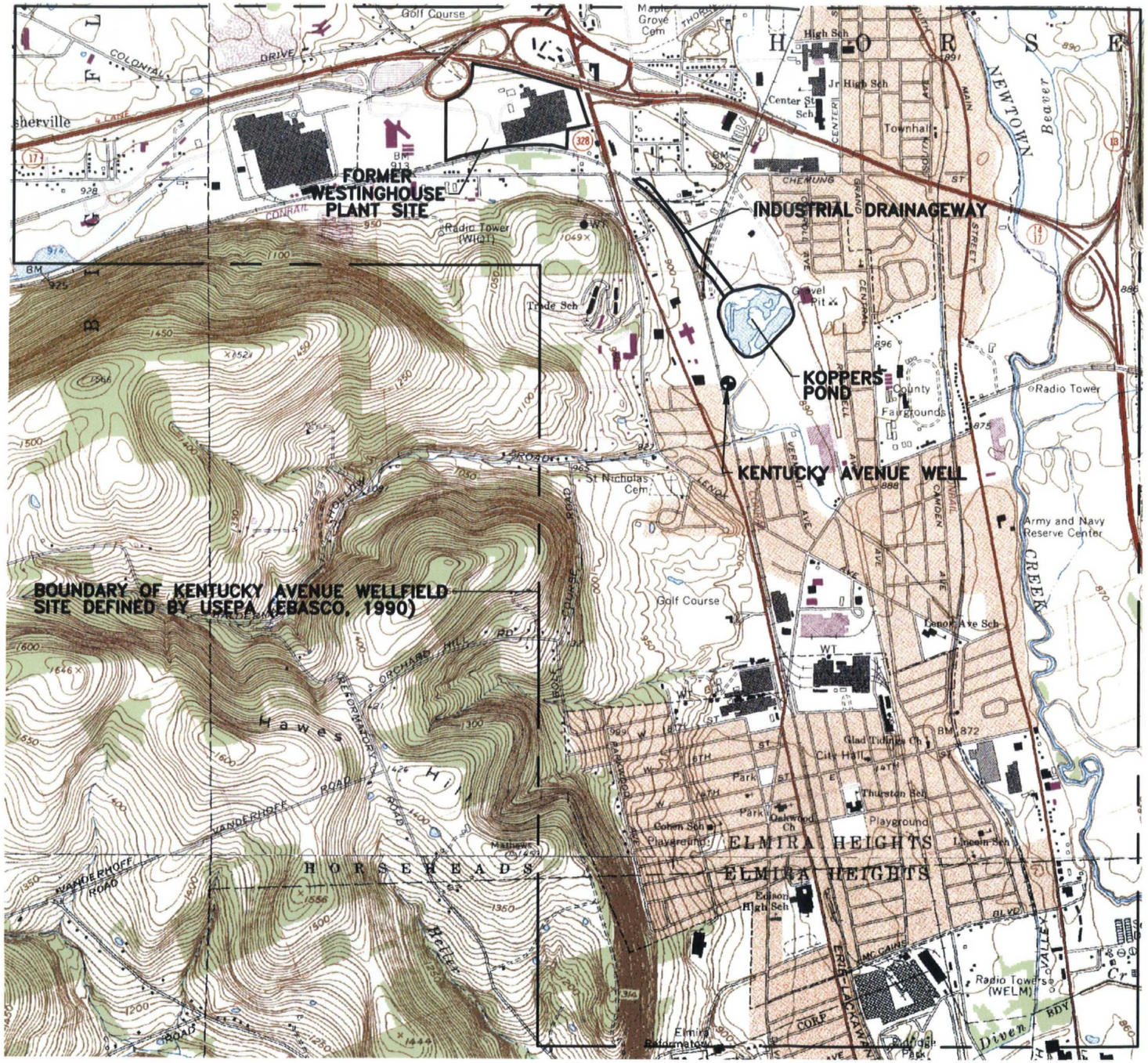
(d) Samples to be analyzed sequentially. The numbers of actual QC samples will be prorated according to the actual number of field samples.

(e) Select sediment samples (up to six) will be analyzed for grain size analysis and for acid volatile sulfide/simultaneously extracted metals (AVS/SEM). The locations selected for grain-size and AVS/SEM analyses may or may not correspond.

(f) If upon field inspection collected fish specimens show deformities that may be indicative of PAH impacts, selected fish tissue samples will also be analyzed for TCL SVOCs.

FIGURES

DRAWING NUMBER 07502B1



SCALE

0 2000 4000 FEET

QUADRANGLE LOCATION

REFERENCE:

MODIFIED FROM U.S GEOLOGICAL SURVEY HORSEHEADS, NEW YORK, AND ELIMIRA, NEW YORK–PENNSYLVANIA, QUADRANGLES, PHOTOREVISED 1978.

FIGURE 1

SITE LOCATION MAP

KENTUCKY AVENUE WELLFIELD SITE – OU4
HORSEHEADS, NEW YORK

PREPARED FOR

KOPPERS POND RI/FS GROUP

CUMMINGS RITER CONSULTANTS, INC.

DRAWING NUMBER 07502B1

DRAWN BY: T.E. McKee	DATE: 1-31-07
CHECKED BY: W.C. Smith	DATE: 2-19-07
APPROVED BY: W.C. Smith	DATE: 2-19-07

PLOT SCALE:

FIGURE 2
SITE PLAN
KENTUCKY AVE. WELLFIELD SITE - OU4
HORSEHEADS, NEW YORK

CUMMINGS RITER CONSULTANTS, INC.

Prepared For:
KOPPERS POND RI/FS GROUP

hheads-1.mxd

Approx. Property Line

Property lines and owners based on Chemung Co. Tax Maps.

Approx. Pond Sample Location

- CDM (1998)
- Phillips (1994-95)
- CEC (2003)

0 250 Feet

hheads-2.mxd

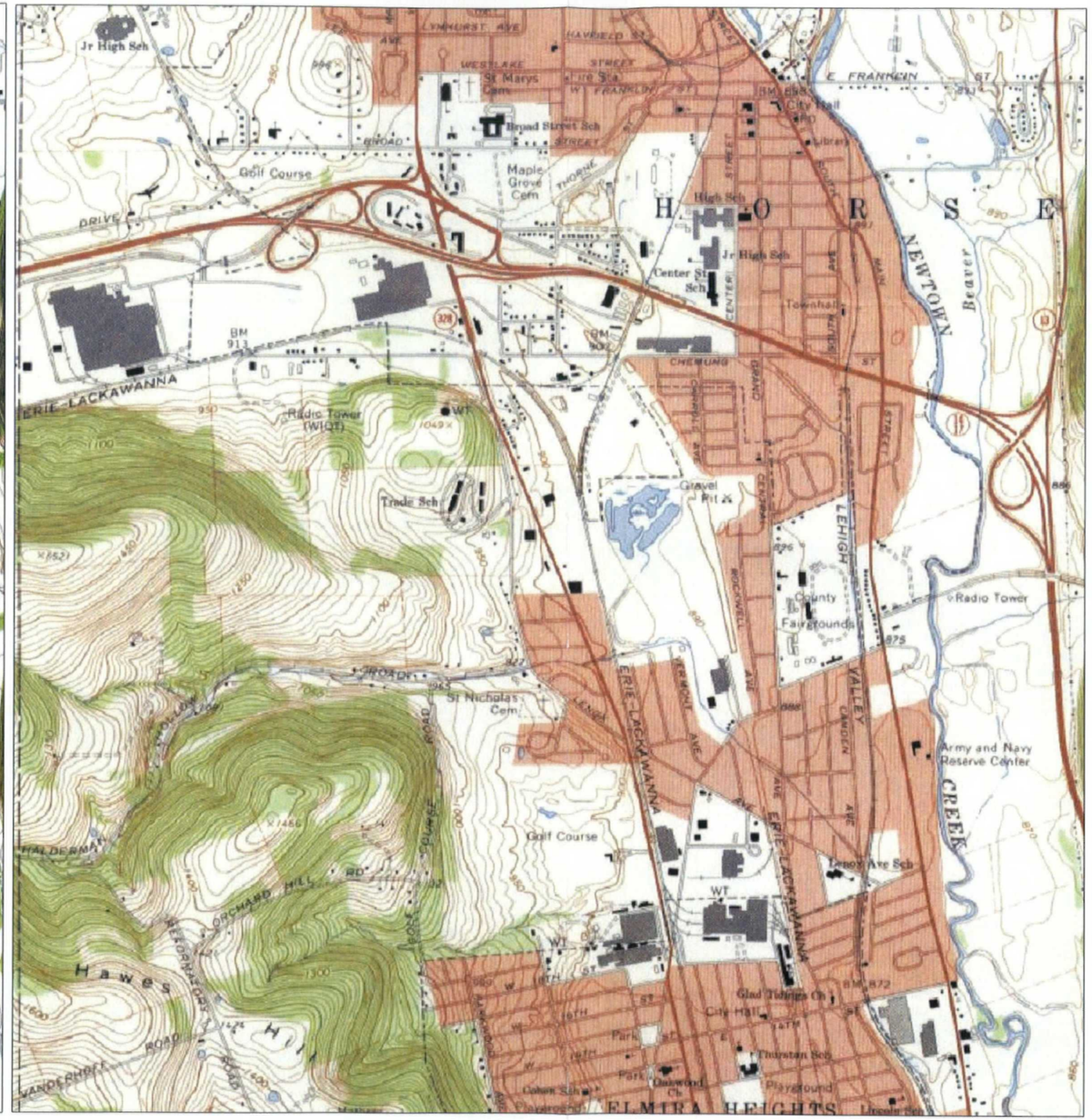
Prepared For:
KOPPERS POND RI/FS GROUP

CUMMINGS RITER CONSULTANTS, INC.

FIGURE 3
HISTORICAL POND SAMPLING LOCATIONS
KENTUCKY AVE. WELLFIELD SITE - OU4
HORSEHEADS, NEW YORK

DRAWING NUMBER 2B2

1953



1969

REFERENCE: U.S. GEOLOGICAL SURVEY, HORSEHEADS, NEW YORK QUADRANGLE, DATED AS INDICATED.

APPROXIMATE SCALE

0 2000 4000 FEET

PLOT SCALE: 1

CUMMINGS RITER CONSULTANTS, INC.
10 Duff Road
Suite 500
Pittsburgh, PA 15235
(412) 241-4500
Fax: (412) 241-7500

REVISIONS			
REV.	DESCRIPTION	DATE	APPROVED

DRAWN BY:	B.MAURER	DATE: 2-2-07
CHECKED BY:	W.C. Smith	DATE: 2-19-07
APPROVED BY:	W.C. Smith	DATE: 2-19-07

FIGURE 4

HISTORICAL TOPOGRAPHIC MAPS

KENTUCKY AVENUE WELLFIELD SITE - OU4
HORSEHEADS, NEW YORK

PREPARED FOR

KOPPERS POND RI/FS GROUP

SIZE	DRAWING NUMBER	REV.
B	07502B2	

SCALE: SHOWN SHEET ____ OF ____

DRAWING NUMBER 07502E2V1

SB-8A

OLD HORSEHEADS LANDFILL

SB-9A

SB-6A

MW-112S

MW-103S

MW-103D

KOPPERS POND

CW-10D

CW-10S

MW-106D

CW-9S

CW-9D

MW-108D

LEGEND

MW-112S ▲ MONITORING WELL LOCATION

SB-9A ◐ SOIL BORING LOCATION

ESTIMATED EXTENT OF C. & D. DEBRIS

REFERENCE:

1. MAPPING COMPLETED BY STEREOPHOTO GRAMMETRIC METHODS BY WEILER ASSOCIATES FROM 1:4800 SCALE AERIAL FLOWN 11-09-91.
2. EXTENT OF C. & D. DEBRIS LINE AND SOIL BORING LOCATIONS WERE TAKEN FROM FAGAN ENGINEERS, CLOSURE INVESTIGATION REPORT, DATED MARCH, 1991. DRAWING TITLED "SITE PLAN", FIGURE 1-2.

SCALE

100 0 100 200 FEET

REVISIONS			
REV.	DESCRIPTION	DATE	APPROVED

CUMMINGS RITER CONSULTANTS, INC.
CORPORATE HEADQUARTERS
10 Duff Road
Duff Office Center, Suite 500
Pittsburgh, PA 15235
(412) 241-4500
Fax: (412) 241-7500

FIGURE 5
REPORTED LIMITS OF CONSTRUCTION DEBRIS DISPOSAL, OLD HORSEHEADS LANDFILL

KENTUCKY AVENUE WELLFIELD SITE - OU4
HORSEHEADS, NEW YORK

PREPARED FOR
KOPPERS POND RI/FS GROUP

SIZE	SCALE	REV.	DRAWING NUMBER
E	1" = 100'		07502E2V2

DRAWN BY:	T.N. Fitzroy	DATE: 11-28-07
CHECKED BY:	W.C. Smith	DATE: 12-5-07
APPROVED BY:	W.C. Smith	DATE: 12-5-07

PLOT SCALE: 1=1

DRAWING NUMBER 07502E1

WESTINGHOUSE DISCHARGE LINE

WESTINGHOUSE WASTEWATER TREATMENT PLANT (CLOSED 1996)

TREATED EFFLUENT(NOT USED)

PLUGGED

001W (001CH AFTER 1996)

12" DRAIN LINE

N.Y. ROUTE 17

SW-3

FORMER BOILER BLOWDOWN LINE

002W

BARRIER WELL WATER TREATMENT FACILITY(1996)

SW-6

001W AFTER 1996

CUTLER-HAMMER AND WESTINGHOUSE LINES SEPARATED(1996)

CUTLER-HAMMER

PARKING LOT

DRAIN LINE -D-

IMAGING AND SENSING TECHNOLOGY CORPORATION

IMAGING AND SENSING TECHNOLOGY CORPORATION

TOSHIBA DISPLAY DEVICES

IMAGING AND SENSING TECHNOLOGY CORPORATION

SW-5

TO CHEMUNG STREET OUTFALL

001T

GUARD HOUSE

TOSHIBA DISPLAY DEVICES

TOSHIBA DISPLAY DEVICES

TOSHIBA DISPLAY DEVICES

TOSHIBA DISPLAY DEVICES

STATE ROUTE 328

SW-7

SOUTH PARKING

TOSHIBA WASTEWATER TREATMENT PLANT (CLOSED 2004)

BARRIER WELL BUILDING (RETAINED ASSET)

BW-2

BW-1

NORFOLK SOUTHERN CORPORATION

905

910

915

920

LEGEND

BW-1 ▲	BARRIER WELL LOCATION
SW-5 ◪	EXISTING WATER SUPPLY WELL LOCATION-
	TOPOGRAPHICAL CONTOUR
-D-	WASTEWATER DRAIN LINE
001W ○	PERMITTED OUTFALL
	BARRIER WELL WATER LINE

NOTES:

1. BASE MAP DERIVED FROM BLASLAND, BOUCK & LEE, INC. DRAWING G-1 FILE NUMBER 650.05.25F DATED NOVEMBER 1995.
2. OUTFALL ARRANGEMENT BASED ON FIGURE 1-12 OF PRIMARY SITE CHARACTERIZATION SUMMARY(BURLINGTON ENVIRONMENTAL, JANUARY 1995) DATED 7/29/94. TITLED "WASTE LINE LOCATIONS".
3. LOCATION OF UNDERGROUND UTILITIES AND OTHER UNDERGROUND STRUCTURES OBTAINED BY FIELD MEASUREMENTS WHERE POSSIBLE,.
4. THIS DRAWING DOES NOT DEPICT THE EXISTENCE AND/OR LOCATION OF ALL UNDERGROUND UTILITIES.
5. SEE TABLE 1 AND DISCUSSIONS IN SECTIONS 2.5.1 AND 3.1.1.1 FOR OWNERSHIP, USE, AND DISCHARGE HISTORY.

SCALE
80 0 80 160 FEET

PLOT SCALE: 1"=

REVISIONS			
REV.	DESCRIPTION	DATE	APPROVED

CUMMINGS RITER CONSULTANTS, INC.
CORPORATE HEADQUARTERS
10 Duff Road
Duff Office Center, Suite 500
Pittsburgh, PA 15235
(412) 241-4500
Fax: (412) 241-7500

FIGURE 6
DISCHARGE LOCATIONS FORMER WESTINGHOUSE HORSEHEADS PLANT SITE

HORSEHEADS, NEW YORK

PREPARED FOR
KOPPERS POND RI/FS GROUP
HORSEHEADS, NEW YORK

SIZE	SCALE	REV.	DRAWING NUMBER
E	1" = 80'	—	07502E1

DRAWN BY:	T.N. Fitzroy	DATE: 6-14-07
CHECKED BY:	W.C. Smith	DATE: 12-5-07
APPROVED BY:	W.C. Smith	DATE: 12-5-07

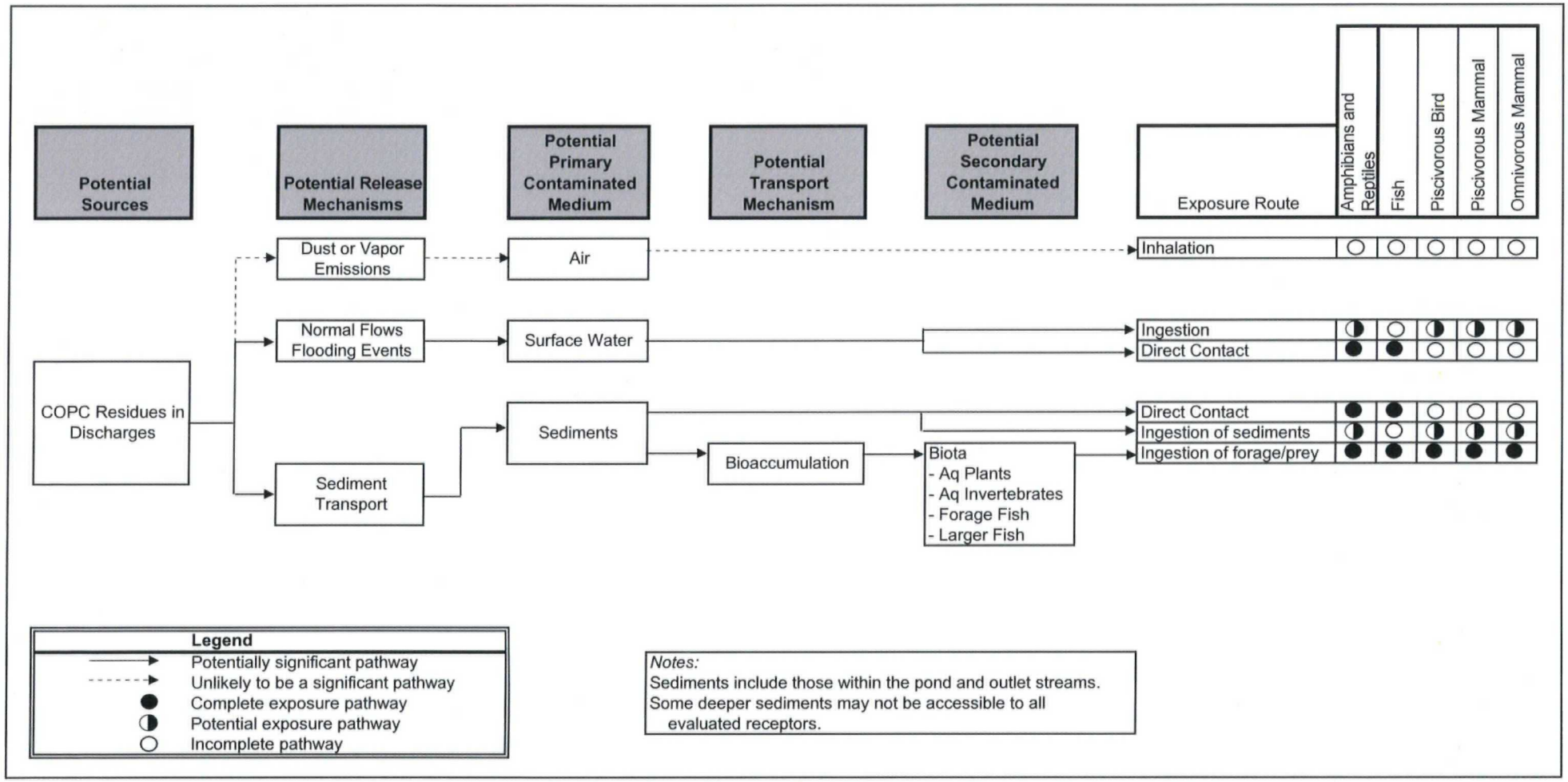


Figure 7. Generic Conceptual Site Model for the Koppers Pond Supplemental Baseline Ecological Risk Assessment

Proposed Pond Sample Location

Proposed Outlet Channel Sample Location

0 200 400 Feet

hheads-3.mxd

Prepared For:
KOPPERS POND RI/FS GROUP

CUMMINGS RITER CONSULTANTS, INC.

FIGURE 8
PROPOSED POND AND OUTLET CHANNEL SAMPLING LOCATIONS
KENTUCKY AVE. WELLFIELD SITE - OU4
HORSEHEADS, NEW YORK

FIGURE 9
POTENTIAL ONGOING SOURCES INVESTIGATION
KENTUCKY AVE. WELLFIELD SITE - OU4
HORSEHEADS, NEW YORK

CUMMINGS RITER CONSULTANTS, INC.

Prepared For:
KOPPERS POND RI/FS GROUP

hheads-5.mxd

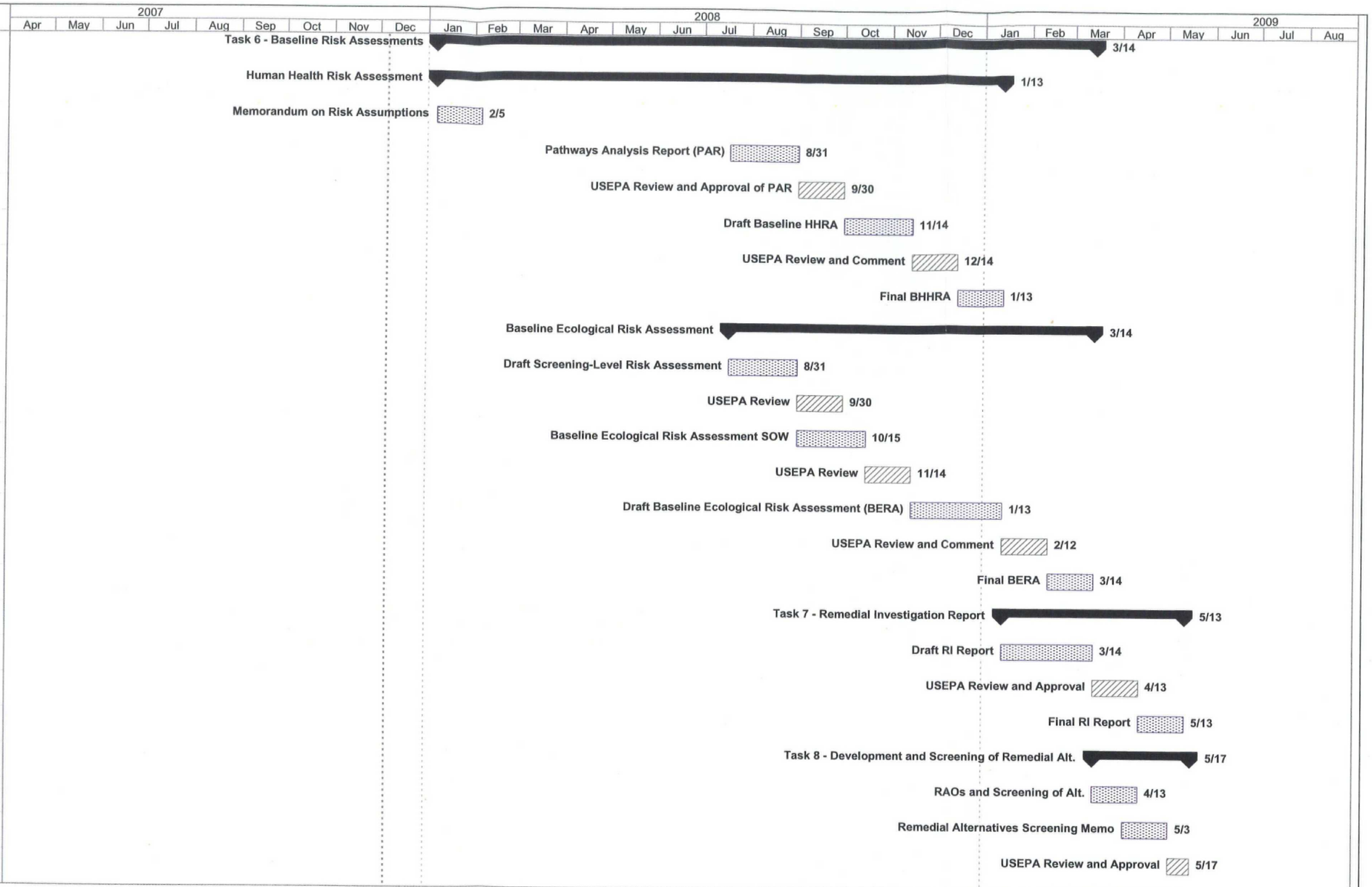
ID	Task Name
1	**Preliminary Conceptual Site Model**
2	**Task 1 - RI/FS Work Plan**
3	Work Plan Preparation and Submittal
4	USEPA Review
5	Work Plan Revisions
6	USEPA Approval
7	**Task 2 - Community Relations Plan**
8	Plan Preparation and Submittal
9	USEPA Review and Approval
10	Site Mailing List
11	RI/FS Commencement Fact Sheet
12	**Task 3 - Site Characterization**
13	Prefatory Activities
14	Tolling Period for Winter Weather
15	Field Investigation
16	Data Analysis
17	Data Validation and Management
18	Draft Site Characterization Summary Report
19	USEPA Review and Comment
20	Final Site Characterization Summary Report
21	Fate and Transport Model Memorandum (If Necessary)
22	**Task 4 - Identification of Candidate Technologies**
23	Identification of Candidate Technologies Memo
24	USEPA Review and Approval
25	**Task 5 - Treatability Studies (As Necessary)**

ised Koppers Pond RI/FS Project Schedule
Revised RIFS Schedule.mpp
Date: Thu 12/6/07

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks | Group By Summary
Progress | Summary | Rolled Up Milestone | Split | Project Summary | Deadline

FIGURE 10, 1 of 3

ID	Task Name
26	**Task 6 - Baseline Risk Assessments**
27	**Human Health Risk Assessment**
28	Memorandum on Risk Assumptions
29	Pathways Analysis Report (PAR)
30	USEPA Review and Approval of PAR
31	Draft Baseline HHRA
32	USEPA Review and Comment
33	Final BHHRA
34	**Baseline Ecological Risk Assessment**
35	Draft Screening-Level Risk Assessment
36	USEPA Review
37	Baseline Ecological Risk Assessment SOW
38	USEPA Review
39	Draft Baseline Ecological Risk Assessment (BERA)
40	USEPA Review and Comment
41	Final BERA
42	**Task 7 - Remedial Investigation Report**
43	Draft RI Report
44	USEPA Review and Approval
45	Final RI Report
46	**Task 8 - Development and Screening of Remedial Alt.**
47	RAOs and Screening of Alt.
48	Remedial Alternatives Screening Memo
49	USEPA Review and Approval

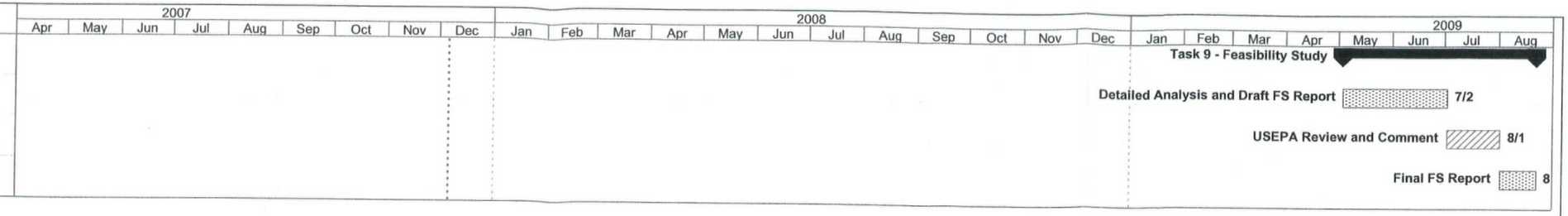


[redacted]sed Koppers Pond RI/FS Project Schedule
[redacted]Revised RIFS Schedule.mpp
Date: Thu 12/6/07

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks | Group By Summary
Progress | Summary | Rolled Up Milestone | Split | Project Summary | Deadline

FIGURE 10, 2 of 3

ID	Task Name
50	**Task 9 - Feasibility Study**
51	Detailed Analysis and Draft FS Report
52	USEPA Review and Comment
53	Final FS Report



ed Koppers Pond RI/FS Project Schedule
Revised RIFS Schedule.mpp
Date: Thu 12/6/07

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks | Group By Summary
Progress | Summary | Rolled Up Milestone | Split | Project Summary | Deadline

FIGURE 10, 3 of 3

APPENDIX A

SAMPLING AND ANALYSIS PLAN / VOLUME I - FIELD SAMPLING PLAN

CUMMINGS RITER

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APPENDIX A
SAMPLING AND ANALYSIS PLAN
VOLUME I – FIELD SAMPLING PLAN
KOPPERS POND
KENTUCKY AVENUE WELLFIELD SUPERFUND SITE
OPERABLE UNIT 4
HORSEHEADS, NEW YORK

1.0 INTRODUCTION

On behalf of the Koppers Pond RI/FS Group (the Group), Cummings/Riter Consultants, Inc. (Cummings/Riter), with assistance from AMEC Earth & Environmental, Inc. (AMEC), has prepared this Sampling and Analysis Plan (SAP) to support remedial investigation (RI) activities to be conducted for Koppers Pond in the Village and Town of Horseheads, Chemung County, New York (the Site). Figure 1 of the Remedial Investigation/Feasibility Study (RI/FS) Work Plan shows the Site location. Pursuant to the Administrative Settlement Agreement and Order on Consent (Index No. CERCLA-02-2006-2025), Koppers Pond is being addressed as Operable Unit 4 of the Kentucky Avenue Wellfield Superfund Site. Data obtained through completion of the RI will be used to characterize environmental conditions, evaluate potential human health and ecological risks, and, if unacceptable risks are identified, support the evaluation of potential remedial action alternatives in the FS.

The SAP consists of two plans: the Field Sampling Plan (FSP) and the Quality Assurance Project Plan (QAPP). Appendix A is the FSP and is to be used in conjunction with the QAPP (Appendix B) to support activities related to the performance of the RI/FS. The SAP has been prepared in accordance with U.S. Environmental Protection Agency (USEPA) *Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA* (EPA 540/G-89/004, October 1988). The objective of this FSP is to describe sampling procedures to be followed during field activities conducted in support of the RI/FS for the Site.

Koppers Pond consists of an approximately eight-acre, "V" shaped, warm water pond with typical water depths of approximately three to six feet. At normal stage, the surface water elevation is at 887± feet above mean sea level. The pond receives inflow at the northern end of its western leg from the Industrial Drainageway, a surface water course that originates at the outlet of a 74-inch diameter underground pipe (Chemung Street Outfall) approximately 2,300 feet to the northwest. The Industrial Drainageway receives permitted process discharges originating at the former Westinghouse Electric Corporation (Westinghouse) Horseheads plant site and surface runoff from a contributory watershed area of approximately 604 acres. Discharge from Koppers Pond flows into two outlet streams at its southern end, which converge approximately 500 feet downstream to form the outlet channel. The flow in this outlet channel eventually converges with Halderman Hollow Creek, which in turn feeds into Newtown Creek, a primary tributary to the Chemung River.

As described in Section 6.3.1 of the RI/FS Work Plan, the major RI tasks and the field and data collection activities associated with these tasks are as follows:

- Task 3.1 - Surveying and Mapping
  - Establish survey control
  - Install pond staff gauge

- Task 3.2 - Surface Water and Sediment Sampling
  - Collection of pond and outlet channel surface water samples
  - Collection of pond and outlet channel sediment samples

- Task 3.3 - Pond Bathymetry
  - Conduct pond bathymetry survey (navigable portion) using Global Positioning System (GPS) and echo sounder
  - Collect pond depth measurements manually in shallows and other non-navigable areas

- Task 3.4 - Assess Sediment Thickness
  - Measure sediment thickness at each sediment sampling location
  - Collect additional measurements if sediment thickness is non-uniform

- Task 3.5 - Assess Potential Ongoing Sources
  - Collect surface water samples and flow readings of the barrier well treated water discharge and the Cutler-Hammer Division of Eaton Corporation (Cutler-Hammer) discharge at the former Westinghouse Horseheads plant site
  - Conduct video survey of underground piping upstream of the Chemung Street Outfall
  - Collect sample of floc if present in underground piping
  - Collect a surface water sample of the Industrial Drainageway at the Chemung Street Outfall
  - Perform field reconnaissance of potential storm water inflows to the Industrial Drainageway and Koppers Pond
  - Collect samples of any identified significant points of storm water inflow
  - Inspect north shore of pond and northeast bank of lower drainageway adjacent to the Old Horseheads Landfill for visual indications of seeps
  - Sample any identified seeps associated with the Old Horseheads Landfill and draining to Koppers Pond

- Task 3.6 - Assess Pond Hydrology
  - Collect measurements of pond surface elevation
  - Collect measurements of nearby groundwater elevations

- Task 3.7 - Fish Tissue Sampling
  - Collect fish specimens for laboratory analysis

In addition, local water bodies exhibiting physical characteristics similar to those of Koppers Pond, but not potentially affected by Site sources, will be identified as candidate reference ponds. The need for investigation of such reference ponds (e.g., sediment sampling, fish tissue sampling) will be determined following the review of RI data and the comparison of these data to prior sampling results for Koppers Pond.

It is anticipated that AMEC will perform the fish tissue sampling. Fagan Engineers, P.C. of Elmira, New York will perform some of the surveying tasks, provide technical and logistical support for field activities, and oversee a specialty contractor in the performance of the video surveying of the Chemung Street Outfall. Cummings/Riter will perform the balance of RI field activities. The following sections describe the procedures to be followed for these activities.

2.0 PROJECT APPROACH AND SAMPLING OBJECTIVES

The primary objectives for the RI are to gather representative environmental data to allow for a detailed evaluation of Site-related impacts in potentially affected media and of cross-media impacts, support risk assessment, and support the selection of an appropriate remedial alternative, if needed, for the Site. RI data will be used to refine and enhance the previously conducted human health and ecological risk assessments, as needed. Site investigation activities may also be necessary before the identification and evaluation of remedial action alternatives can be completed.

2.1 SURVEYING AND MAPPING

Existing survey control will be reviewed and evaluated to determine if additional control is necessary for completion of the RI/FS tasks. If additional survey control is needed, such control will be established in the vicinity of Koppers Pond and tied to State Plane Coordinates and North American Vertical Datum of 1988 (NAVD 88).

Topographic mapping of the Koppers Pond area is available from Operable Unit 2 activities. This mapping is of suitable horizontal scale (1 inch = 50 feet) and contour interval (1 foot) for use as the base map for Operable Unit 4 field investigations. The existing mapping is suitable for delineation of areas of potential seasonal flooding and various habitats and for definition of surface water features.

2.2 SURFACE WATER AND SEDIMENT SAMPLING

Sediment samples will be collected to evaluate the current distribution of constituents of potential concern (COPCs) in Koppers Pond and the outlet channels. Surface water samples will also be collected from the pond and outlet channel to obtain current water quality data. Surface water and sediment analytical results will be used to compare current conditions to those previously reported, assess risks to potential receptors, and, as needed, evaluate potential remedial action alternatives.

2.3 POND BATHYMETRY

The pond bottom will be mapped using proven bathymetric survey methods to establish the water depth and to help establish the available habitats for potential ecological receptors. This information will also be used to support evaluations of the biological productivity of the Site, estimate sediment volumes, and evaluate the effectiveness of applicable response actions, if necessary.

2.4 SEDIMENT THICKNESS

Sediment thickness in Koppers Pond will be estimated by manual probing at select locations in areas accessible by boat or by wading. Sediment thickness measurements will be taken throughout the pond, including at each sediment sampling location. Sediment thickness will be used to estimate sediment volumes, determine the number of sediment samples at sampling locations, and assess depositional patterns within the pond.

2.5 EVALUATION OF POTENTIAL ONGOING SOURCES

There are several potential sources of ongoing impacts to Koppers Pond, including permitted (point source) discharges, non-regulated point source discharges, and non-point source discharges (i.e., runoff from impacted soils and possible seeps from the Old Horseheads Landfill into the Industrial Drainageway). Potential sources will be identified by reconnaissance of the Industrial Drainageway, the pond area, outlets, outlet channel, and video survey of the Chemung Street Outfall pipe. Samples of concentrated flows will be collected, including flow from the Chemung Street Outfall, culverts crossing beneath the Norfolk Southern Corporation railroad tracks, and other potential sources to evaluate continuing contributions of COPCs to the Site. In addition, floc adhering to the Chemung Street Outfall pipe, if observed, will be sampled.

2.6 POND HYDROLOGY

The hydrology of Koppers Pond will be studied to assess the interaction of local groundwater and surface water. Because of the barrier well discharge and other surface water inflows, the pond typically recharges groundwater, although the pond may receive groundwater discharges under certain circumstances. Groundwater and surface water elevations will be measured for a period of three months by installing transducers with data loggers in the pond and in existing groundwater monitoring wells proximate to

Koppers Pond. An understanding of groundwater and surface water interaction will contribute to the understanding of fate and transport mechanisms affecting the COPCs associated with Koppers Pond.

2.7 FISH SAMPLING

The objective of the fish survey is to collect fish for evaluation of risks to human and ecological receptors. Fish sampling will be conducted after consideration of the sampling protocols described in relevant USEPA and other guidance, including the following:

- *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories* (Volume 1) (USEPA, November 2000); and
- *Procedures for Collection and Preparation of Aquatic Biota for Contaminant Analysis* (New York State Department of Environmental Conservation [NYSDEC], October 2002).

The survey will also be conducted in accordance with conditions set forth in the New York Scientific Collector's Permit.

Although it is difficult to ensure quantitatively the outcome of the pending fish collection efforts, the following targets for these collections will provide relevant data for both the human and ecological risk assessments:

- Collect 10 individual carp of one size class (10 to 13 inches);
- Collect 10 individual fish of similar-sized sunfish or crappie (8 to 10 inches); and
- Collect two types of composites of forage fish/minnows: three composites of smaller forage fish (30 to 100 millimeters [mm]) and three additional composites of larger forage fish/minnows (100 to 300 mm).

Along with fish sampling, qualitative data of the available fish habitat will be collected by measuring the following:

- Assessment of in-pond cover (e.g., large woody debris, root wads, root mats, undercut banks, gravel bars, and macrophytes),
- Floodplain and land use around the pond, and
- Degree of canopy cover.

The fish habitat assessment is a qualitative tool to be used in conjunction with other data (e.g., surface water quality, sediment quality, fish examination) in the overall evaluation of the pond ecosystem and stressors that might affect populations of various fish species. This habitat assessment will also be used in conjunction with other data to develop an estimate of potential and sustainable fish populations and the yield of the pond for (human) edible fish.

3.0 CONSTITUENTS OF POTENTIAL CONCERN

Based on the results of sediment and surface water samples collected during previous investigations, the Preliminary Conceptual Site Model identified COPCs for surface water as consisting of metals and the pesticide compounds α-BHC (benzene hexachloride) and β-BHC. Metals and hydrophobic organic compounds, such as polychlorinated biphenyls (PCBs), pesticides, and polynuclear aromatic hydrocarbons, have been identified as the COPCs for sediment.

Discussion of COPCs in sediment, surface water, and potential ongoing sources is provided in Section 4.0 of the RI/FS Work Plan. As described in the RI/FS Work Plan, conditions in Koppers Pond are dynamic, and certain aspects and characteristics of the pond have likely changed since the time data were collected as part of prior studies. Accordingly, the RI for Koppers Pond is primarily focused on collecting current information regarding surface water and sediment quality and comparing these data to the results of previous studies. The RI will examine a wide suite of organic and inorganic constituents to identify the COPCs currently associated with Koppers Pond that may potentially cause or contribute to unacceptable human health or ecological risks. Analytical parameters for all environmental media, as well as for fish tissue samples, are set forth in the QAPP. Analyses will be conducted using USEPA-approved methods.

4.0 SAMPLING LOCATIONS AND FREQUENCY

This section discusses the selection of sampling locations and frequency of sample collection. Table A-1 outlines the proposed sampling approach and presents the number (and type) of samples that will be collected during the RI at the Site, including quality control (QC) samples. Figure A-1 provides the locations of the proposed surface water and sediment samples.

4.1 SURFACE WATER MEASUREMENTS

A staff gauge will be placed in the open water of Koppers Pond to facilitate measurements of the surface water elevation. The staff gauge will be placed at a location where the pond bottom is below the historical low water elevation and can be read from shore. After installation, the staff gauge will be surveyed to tie elevations to NAVD 88. A minimum of 12 staff gauge readings of surface water elevation using the staff gauge will be used for verification of the transducer measurements of pond levels (Section 4.2).

4.2 POND HYDROLOGY

Groundwater elevations and surface water elevations will be automatically recorded four times daily for a period of three months. One transducer will be placed in an area of the pond bottom that is below the historical low water elevation. In addition, transducers will be placed in five existing groundwater monitoring wells proximate to Koppers Pond (e.g., MW-112S, CW-9S/9D, and CW-10S/10D). Data loggers will be used to record transducer measurements, and data will be downloaded monthly.

4.3 BATHYMETRIC SURVEY

A bathymetric survey will be performed by boat in navigable portions of the pond or by wading where the water is shallow and the pond bottom is sufficiently firm. It is preferable that the bathymetric survey be performed while the pond level is high to facilitate navigation by boat. The bathymetric survey will be conducted using random track lines, as opposed to pre-defined cross-section lines. The boat will traverse the pond with depth soundings taken every 25 to 50 feet along the traveled route. Track lines will

be monitored using GPS techniques to ensure adequate spatial coverage of the pond. This survey technique maximizes data collection and avoids the time delays associated with trying to align the survey boat along predetermined grid lines.

Upon completing the bathymetric survey, the data will be post-processed to calculate elevations tied to Site control points.

4.4 SEDIMENT THICKNESS

Sediment thickness in the pond will be measured at each of the 13 sediment sampling locations (shown on Figure A-1), as well as additional locations as needed to provide sufficient data to estimate sediment volumes. A probe, consisting of a hollow metal pipe with a metal plug at the advancing end, will be manually advanced in navigable portions of the pond into the sediment until refusal is reached in the sand and gravel deposits underlying the sediment. The refusal elevation will be used with the bathymetric survey data to estimate the sediment thickness at each location. The sediment thickness data will also be used to determine how many vertically discrete sediment samples will be collected at each location.

4.5 SURFACE WATER AND SEDIMENT SAMPLING

Surface water and sediment samples will be collected and analyzed to characterize potential impacts to these media from COPCs. The following locations are proposed for sample collection during the RI:

- **POND** – Thirteen locations approximately evenly spaced throughout the pond to provide sufficient coverage to allow comparisons to previously collected data and investigate the range of hydraulic conditions present in the pond (e.g., center channels, near-shore shallows); and

- **OUTLET CHANNELS** – One in the East Outlet, one in the West Outlet, and two in the downstream Outlet Channel.

Sample locations in the pond, outlets, and outlet channel are shown on Figure A-1. The sampling strategy is to collect a discrete vertical sample to represent the uppermost six inches of sediment and deeper samples as needed to be representative of each additional

12 inches of sediment. The sediment thickness data (Section 4.4) will be used to determine how many vertically discrete sediment samples will be collected at each location. If sediments are thicker than the anticipated 24 inches, more samples will be collected. If sediments are less than 24 inches thick, fewer samples will be collected.

Sample locations are approximately evenly spaced throughout the pond to provide sufficient coverage to allow comparisons to previously collected data and investigate the range of hydraulic conditions present (e.g., center channels, near-shore shallows). The proposed sediment sample location should be sufficient to assess the different depositional characteristics throughout the pond. Variations in sediment depth may reflect variations in hydraulic conditions.

Surface water samples will be analyzed for target compound list (TCL) organics; target analyte list (TAL) inorganics; and the general chemistry parameters ammonia, fluoride, hardness, nitrites, and total suspended solids. Dissolved oxygen, pH, oxidation-reduction potential (ORP), temperature, and specific conductance will be measured in the field at the time of sampling. Aqueous samples collected for TAL analysis will be analyzed for both the total and dissolved fractions of metals to allow for direct comparison of results to ambient water quality criteria.

Sediment samples will be analyzed for TCL organics, TAL inorganics, and total organic carbon. Field measurements of pH and ORP will also be made at the time of sampling. Select samples from the pond and outlet channels will be tested for grain-size distribution, and select sediment samples collected from the first (0- to 6-inch) interval will be analyzed for acid volatile sulfide/simultaneously extracted metals (AVS/SEM).

The selection of sediment samples for grain-size determination will be based on visual inspection of samples collected in the field with the objective of evaluating the range of sediment materials present in the pond and its outlet channels. It is anticipated that all or nearly all of the pond samples will contain predominantly silt and clay-sized materials, although there may be some coarsening with depth. The outlet channel samples are expected to exhibit a wider range of grain size.

Up to six surface (0- to 6-inch) sediment samples will be analyzed for AVS/SEM to provide insights on the bioavailability of metals. AVS/SEM analysis will be performed following methods described in the USEPA (December 1991) guidance, *Draft Analytical Method for the Determination of Acid Volatile Sulfide in Sediment* (EPA 821/R-91-100). Sample locations for AVS/SEM analysis will include the northwestern area of Koppers Pond where metals concentrations in sediments are expected to be highest and where past studies had indicated sediment toxicity.

4.6 SAMPLING OF ONGOING SOURCES

Figure A-2 depicts possible ongoing sources of COPCs to Koppers Pond and outlets that are currently known. Samples may be collected from such potential sources as described below.

4.6.1 Chemung Street Outfall Pipe

A video survey of the underground pipe terminating at the Chemung Street Outfall will be performed to provide information on pipe alignment, integrity, floc accumulations on pipe walls, and potential sources, other than the discharges from the former Westinghouse Horseheads plant site, that tie into the pipe. The New York State Department of Transportation (NYSDOT) will be contacted to research as-built information on the pipe traversing past the former Westinghouse plant to the Chemung Street Outfall. The Village of Horseheads and the Southern Tier Central Regional Planning data base will also be researched related to storm sewer systems that contribute flow to the discharge from the Chemung Street Outfall.

If floc accumulations are observed within the underground piping, samples will be collected for laboratory analysis of TCL organics and TAL inorganics (see QAPP Section 2.2.1 and Table B2-2). In addition to floc sampling, water samples will be collected of the following:

- Barrier well treated water discharge,
- Cutler-Hammer discharge,
- Significant storm water inflows upstream of the Chemung Street Outfall (if accessible), and
- Chemung Street Outfall.

These water samples will be analyzed for TCL organics, TAL inorganics, and the general chemistry parameters ammonia, fluoride, hardness, nitrites, and total suspended solids. Dissolved oxygen, pH, ORP, temperature, and specific conductance will be measured in the field at the time of sampling.

4.6.2 Storm Water Runoff

Sources of significant storm water runoff that enter the Industrial Drainageway downstream of the Chemung Street Outfall or directly flow into Koppers Pond will be investigated. A field reconnaissance will be conducted of the study area to identify potential sampling locations, including road culverts, culverts under the railroad, and areas receiving runoff from industrial property.

4.6.3 Landfill Seepage

The northern shore of the pond and east bank of the lower reach of the Industrial Drainageway abut the Old Horseheads Landfill. These areas will be inspected for the presence of seeps that may drain into the pond (Figure A-2). Because the northern shore of the pond is relatively flat, seeps are not expected in this area. The east bank of the lower drainageway is steeper, and seeps, if present, are more likely to occur in this area. If seeps are found, they will be sampled and analyzed for TCL organics, TAL inorganics, and the general chemistry parameters ammonia, fluoride, hardness, nitrites, and total suspended solids. Dissolved oxygen, pH, ORP, temperature, and specific conductance will be measured in the field at the time of sampling. Flow rates will be estimated based on field observations.

4.7 Fish Sampling

Based on results of the 1995 and 2003 fish sampling events, common carp, white sucker, largemouth bass, pumpkinseed, black crappie, and green sunfish are likely fish species to be found inhabiting the pond. Although it is difficult to accurately predict the success of the pending fish collection efforts, the plan is to collect two species of fish that are of "recreational size" to be used in the human health evaluation. The target species and size ranges are carp of about 10 to 13 inches (250 to 330 mm) in length, and sunfish or crappie of about 8 to 10 inches (200 to 250 mm) in total length. These may be adjusted

based on the individuals and species collected during sampling. Body lengths will be recorded as total lengths (in mm), consistent with USEPA (February 1995) and NYSDEC (October 2002) guidance documents.

While USEPA (November 2000) recommends that whole fish should be analyzed to mirror the way some consumers may prepare the fish (e.g., stew or soup), skin-on fillets with the belly flap included are often used to more closely portray the standard filleting method used by recreational fishermen (NYSDEC, October 2002). Accordingly, individual skin-on fillets with belly flap will be used for chemical and lipid analysis.

Smaller fish (e.g., minnows, sunfish) that might be preyed upon by the selected, higher trophic level ecological receptors will be collected for the ecological risk assessment. Whole body smaller fish will be collected in sufficient number and weight to provide a sufficient mass for laboratory analysis. Two size classes will be targeted: smaller forage fish/minnows (30 to 100 mm) and larger forage fish/minnows (100 to 300 mm). Composites will likely be required to ensure sufficient sample mass for chemical analysis. Three composites each of the smaller and larger forage fish will be collected.

The numbers of fish described above for collection assume that the species and size class of fish are, in fact, present in Koppers Pond, and will be collected. If other species are collected (e.g., forage species such as shiner), they may substitute or augment the collections described above.

Fish samples will be analyzed for TCL pesticides/PCBs and TAL inorganics. In addition, the collected fish will be examined in the field for any external signs of deformities, tumors, or lesions. If such deformities are noted, suggesting the potential for Polycyclic Aromatic Hydrocarbon (PAH) impacts, equal numbers of individual fish showing deformities, tumors, or lesions and individual fish without deformities will be analyzed for TCL semivolatile organic compounds. The lipid content of fish samples will be determined to facilitate the evaluation of the concentrations of lipid-soluble constituents (e.g., PCBs).

5.0 EQUIPMENT AND PROCEDURES

This section describes equipment and procedures to be used during the performance of field activities conducted as part of RI. The Standard Operating Procedures (SOPs) for the field instruments to be used during the performance of the RI field activities including calibration procedures for these field instruments are included in Attachment A-1.

5.1 GENERAL DOCUMENTATION REQUIREMENTS

Each day work is performed at the Site, a field activity daily log will be completed by the field staff. It will be the responsibility of the members of the field crew to ensure that this record is completed. Information to be provided on the log includes, as appropriate, the following:

- Field activity subject,
- General work activity,
- Unusual events,
- Changes to plans and specifications,
- Visitors on Site,
- Subcontractor progress or problems,
- Communications with USEPA, NYSDEC, the New York State Department of Health, or community members,
- Weather conditions, and
- Personnel on Site.

The field activity daily log will be signed by the individual who prepares it. Field activity daily logs will be submitted on a weekly basis to the Project Supervisor or Project Manager. Following review, the logs will be placed in the project file. A blank field activity daily log is provided in Attachment B-2 of the QAPP (Appendix B).

Other data forms used for documenting sample data and field tests include sample collection and chain-of-custody forms. Field forms are provided in Attachment B-2 of the QAPP.

5.2 WATER LEVEL MEASUREMENTS

The staff gauge for Koppers Pond water level measurements will be graduated in increments of hundredths of a foot. A fixed point on the staff gauge will be marked and surveyed so that gauge readings can be converted to NAVD 88 elevation.

5.3 BATHYMETRIC SURVEY

The bathymetric survey will be performed using an echo sounder to obtain water depth measurements in navigable portions of the pond. The echo sounding device is effective in waters no less than 1.8 feet deep, and has a resolution of approximately 0.1 foot.

The echo sounder will be calibrated by using the portable depth gauge at two locations and adjusting the echo sounder to equate to the manual readings. The manual depth measurement tool is a metal plate mounted on a handle that is pushed downward through the water until the sampler feels resistance. Depth to sediment is measured by the length of submerged handle. The echo sounder will be integrated with differential GPS survey equipment to assign horizontal positions to the depth data. Horizontal positional accuracy will be approximately ±1 foot, and vertical accuracy will be approximately ±0.1 foot. Raw GPS data will be post-processed against fixed base station data to obtain sub-meter accuracy. Once the data are post-processed, horizontal positional accuracy of approximately 1 foot is the result. Site survey control will be used to establish the vertical datum for the survey tied to NAVD 88.

Navigable areas that are too shallow for echo sounding and have solid footing will be manually probed with a portable depth gauge. Water depths in non-navigable portions of the pond will be estimated from shoreline observations or by wading and using the manual depth probe.

5.4 SEDIMENT THICKNESS

Sediment thickness will be measured by manually advancing a metal probe to refusal at select locations, including the 13 sediment sampling locations shown on Figure A-1. Additional thickness measurements will be taken if the sediment thickness is found to be non-uniform. The depth of the sediment bottom will be used with the pond bottom data obtained from the bathymetric survey to estimate the sediment thickness. The length of

the probe at refusal will be measured with a scale in increments of hundredths of a foot. The depth of refusal will be recorded relative to water surface elevation and converted to NAVD 88 elevation.

5.5 SEDIMENT SAMPLING

Sediment samples will be collected in navigable portions of Koppers Pond from a small boat using a 5-centimeter (cm) diameter, 60-cm (about 24-inch) long, Aquatic Research Instruments Russian sediment borer. A description of the sediment borer can be found in Attachment A-1. The borer will be manually pushed to the desired sample depth and rotated clockwise 180 degrees to open the cover plate, expose the sharpened edge of the sample chamber, and collect the sample. To retrieve the sample, the borer is rotated counterclockwise to close the cover plate and contain the sample. The Russian sediment borer was evaluated in a field demonstration by USEPA, and results were published by the *Innovative Technology Verification Report – Sediment Sampling Technology* (December 1999), included in Attachment A-1.

The Russian sediment borer has been shown to preserve sediment stratification in consolidated sediments, but may not preserve stratification in softer, unconsolidated sediments. If pond sediments are found to be unconsolidated, an Aquatic Research Instruments 6.8-cm inside diameter, 60-cm (about 24-inch) long universal percussion corer may be used in lieu of the borer. A description of the corer can be found in Attachment A-1.

Pond sediment samples will be collected in 24-inch depth increments, corresponding to the length of the sampler, with the intent of collecting one discrete grab sample for the uppermost six inches, and deeper samples for each additional 12-inch thickness increment. It is anticipated that up to three samples will be collected at each sample location. If sampler resistance is encountered before reaching the target sampling depth, the sample interval will be truncated. The number of sediment samples may be modified based on the results of field observations.

If sediments are found to be thicker than 24 inches at any location, both the Russian sediment borer and universal percussion corer are suitable for collection of deeper

samples. The Russian sediment borer is lowered to the desired sample depth before rotating clockwise to open the cover plate. Core barrels are available at lengths of up to about eight feet for the universal percussion corer.

Alternatively, a contingency sampling methodology may be employed for collection of sediment samples if sediment thickness exceeds two feet. Two such alternatives have been identified for the contingency approach:

- Push a section of 3-inch diameter solid-wall polyvinyl chloride (PVC) pipe vertically into the sediments to refusal, evacuate the supernatant using a small pump, and sample sediment from the inside of the PVC casing; or

- Fabricate a piston-type sampler using Lexan® tubing, push the sample tube into the sediments to refusal, apply suction to the sample tube, and withdraw the entire sediment core.

Both of these alternate approaches are designed to allow sampling of thick (>24-inch) sediments while maintaining the vertical integrity of the samples. The actual method to be employed (if needed) will be determined from field trials.

Sediment samples will be collected from the outlets and outlet channel at the same locations as the surface water samples, immediately after surface water sampling is complete. Sediment samples in the outlets and channel will be collected from the uppermost six inches of material using a trowel, hand auger, or similar sampling tool. Sample locations will be staked and flagged for future location (if necessary).

Prior to further sample handling, field measurements of pH and ORP will be collected by immersing the meter probe(s) into the wet sediment sample as quickly as possible after collection to ensure minimal changes in these parameters due to mixing or contacting ambient air. With the exception of the volatile organic compound (VOC) and AVS/SEM sample fractions, which will be collected as discrete, non-homogenized grab samples, the sediment removed from the sampler will be divided to represent the depth increments as described above, placed in a stainless-steel bowl, drained of free water, and blended. The blended sediment will then be placed directly into the appropriate sample containers

provided by the analytical laboratory using pre-cleaned, stainless-steel trowels or spoons or new disposable spoons. The VOC fraction of the sediment samples will be collected from a representative portion of the sample interval, drained of free water, and placed directly into the sample containers provided by the laboratory. For the VOC and AVS/SEM samples, the bottles will be filled to the top to ensure no headspace. In addition, the required QC samples will also be collected in accordance with the frequency defined in the QAPP.

Sample collection forms will be completed at each sampling location. A blank sample collection form is provided in Attachment B-2 of the QAPP.

5.6 SURFACE WATER SAMPLING

Surface water samples will be collected by gently submerging a clean laboratory-supplied container (transfer bottle) from approximately mid-depth and filling the appropriate sample bottles. A transfer bottle (or equivalent) will be used so that sample preservatives are not lost during sampling. A coliwasa sampler or long-handled dipper may also be used in place of the transfer bottle for surface water sample collection. Prior to transferring the sample to the laboratory containers and adding any required preservation, field measurements of dissolved oxygen, pH, ORP, temperature, and specific conductance will be collected by immersing the meter probe(s) into the pre-preserved sample. For flowing water, samples will be collected progressively from downstream to upstream locations to minimize sample disturbance.

If a non-aqueous sheen is observed at any of the surface water locations, an additional sample of the water and sheen will also be collected if it is suspected that the sheen is representative of petroleum or other pollutant. In this context, "sheen" refers to a visible, multi-colored film within a discrete area on the water surface suspected to indicate the presence of petroleum or other pollutants. A sheen would be sampled by partially submerging the opening of a clean transfer container within the sheen area.

5.7 POTENTIAL SOURCE AND SEEP SAMPLING

Samples of potential COPC sources to Koppers Pond and pond outlets will be collected by gently submerging a clean laboratory-supplied container (transfer bottle) beneath the water surface and filling the appropriate sample bottles. In collecting seep samples,

special care will be taken to avoid suspended solids in the sample. Samples will be retrieved from approximately mid-depth. If a non-aqueous sheen is observed at any sampling location, an additional sample of the water and sheen will also be collected if it is suspected that the sheen is representative of petroleum or other pollutant. A transfer bottle (or equivalent) must be used so that sample preservatives are not lost during sampling. A coliwasa sampler or long-handled dipper may also be used in place of the transfer bottle for seep sample collection.

5.8 BIOLOGICAL FLOC SAMPLING

Floc samples will be collected by manually scraping the walls of the pipe using a plastic or metal spatula. The scrapings will be collected directly into the sample containers.

5.9 FISH SAMPLING

Electrofishing will be conducted during daylight in the various available habitats (i.e., near and off shore) that exist in Koppers Pond using the appropriate gear (e.g., boat-mounted, backpack, long-line) for the habitat. One sampler will operate and maneuver the boat, and one biologist will operate the electrofishing unit and net stunned fish from the bow of the boat while standing behind a safety railing. Netted fish will be placed into a boat-mounted livewell. The netter will operate a bow-mounted safety foot pedal that controls (on and off) the electric current. In addition, shoreline seining will be conducted in available habitats during daylight to collect forage fish species (e.g., minnows) to supplement those collected during electrofishing. An effort will be made to sample areas that historically have had elevated COPC sediment concentrations.

Fish samples will be identified according to species, weighed (nearest 0.1 gram), and measured (total body length in mm). Fish not retained for tissue analysis will be returned to the pond alive. If present, external evidence of fish disease, tumors, lesions, erosions, fin damage, deformities, and/or skeletal anomalies will be recorded. These data will be recorded on fish sampling field data sheets. Fish vouchering, if necessary, will be conducted in accordance with the conditions outlined in the New York Scientific Collector's Permit.

5.10 SAMPLE PREPARATION AND HANDLING

5.10.1 Sample Containers

For environmental media, sample containers will be supplied by the analytical laboratory as certified pre-cleaned, in accordance with appropriate USEPA guidelines. Sample containers will be filled completely, if possible, to ensure that sufficient sample volume is obtained for laboratory analysis and associated laboratory quality assurance (QA)/QC procedures.

For fish tissue samples, after initial processing to determine species, size, and morphological abnormalities, each fish or composite selected for analysis will be wrapped in hexane-rinsed aluminum foil (per NYSDEC guidance), placed in a food-grade, waterproof plastic bag, and sealed with a label placed on the outside of the bag. A label on the inside of the plastic bag will also be applied because the outer labels often become detached, especially when placed in the coolers with ice.

5.10.2 Sample Preservation and Holding Times

Because certain constituents in water can change chemically with time, it will be necessary to preserve individual samples to maintain the integrity of time-dependent constituents. Laboratory personnel will add the required preservatives to each individual laboratory-supplied sample bottle. Preservation includes maintaining the samples in a chilled condition (4 degrees Celsius [°C]) once they have been collected.

Samples will be stored at 4°C prior to analysis. Fish samples will be cooled immediately after packaging and preserved on wet ice or blue ice packets for shipping to the analytical laboratory within 24 hours. Because filleting has to occur prior to freezing to avoid cross-contamination of ruptured offal, the samples will be shipped to the laboratory in a chilled (not frozen) state. The laboratory will process and prepare the fish tissue samples (e.g., weighing, filleting, homogenizing) in accordance with accepted protocols (e.g., NYSDEC, 2002). Laboratory analysis will be performed within specified holding times to ensure the validity of the analytical results. The type and volume of sample containers, number of containers, preservatives, and holding times for each analytical parameter are provided in Table A-2.

5.10.3 Sample Labeling and Handling

The laboratory will supply blank labels for all sample containers. The labels will be filled out at the time of sample collection by the field personnel performing the sampling. Sample identification is described in Section 6.1. Information marked on the label will include the following:

- Sample identification number,
- Collector's initials,
- Date of collection,
- Type of sample,
- Preservatives used, and
- Analysis to be performed.

The samples will be transported to the laboratory in durable, secured metal or plastic coolers, or laboratory-supplied, insulated shipping containers. Containers will be shipped via common carrier (e.g., Airborne or Federal Express) or hand delivered. Samples will be shipped in accordance with U.S. Department of Transportation and NYSDOT regulations. Chain-of-custody documentation will accompany the samples.

5.11 Field Quality Control Samples

As a check on field sampling QA/QC, trip blanks, equipment rinsate samples, and field duplicates will be prepared and sent to the laboratory at specified frequencies. In addition to the field QA/QC samples, samples will be collected for laboratory QA/QC. These samples consist of matrix spike/matrix spike duplicate (MS/MSD) samples. The frequencies at which these samples will be collected and the numbers of such samples are provided in the QAPP.

A trip blank for liquid samples is a sample bottle filled by the laboratory with analyte-free laboratory reagent water, handled like a sample but not opened, and sent to the laboratory for analysis. Trip blanks are required and analyzed for VOC samples only, and are used to determine if contaminants are introduced during sample handling and shipment. One trip blank will be included with each shipping cooler of VOC samples sent to the laboratory.

Equipment rinsate samples are defined as analyte-free deionized water poured through sampling equipment, transferred to the sample bottle, and then transported to the laboratory for analysis. These samples help determine whether sampling equipment was sufficiently clean before sampling. The equipment rinsate blanks will be analyzed for the same parameters as the sampled media.

A field duplicate is defined as two or more samples collected independently at a sampling location during a single act of sampling. Duplicate samples will be collected by filling alternate sample containers from a homogenized sample from one sample location. For example, a surface water VOC duplicate would be collected by alternately filling the vials for one sample and then into the vials for the duplicate sample. The number of field duplicates required is presented in Table A-1.

Field duplicates will be indistinguishable by the laboratory from other samples. Therefore, one complete sample set will be identified with a "coded" or false identifier in the same format as other identifiers used for this sample matrix. Both the coded and the true identifiers will be recorded on the sample collection form. The coded identifier will be used on the chain-of-custody forms. These coded field duplicates are used to assess the representativeness of the sampling procedure as well as laboratory precision.

MS/MSD samples are required for TCL/TAL analyses of sediment and surface water samples. Samples designated for MS/MSD analysis will be collected at a frequency defined in the QAPP. Double the normal sample volume will be collected for samples selected for MS/MSD analyses. Procedures for collecting MS/MSD samples at any location will be the same as those used to collect field duplicate samples.

5.12 DECONTAMINATION

Decontamination of equipment used for sampling, if not dedicated to a sample, will be carefully performed to minimize any possibility of cross-contamination through the use of tools and equipment. Reusable sampling equipment will be decontaminated prior to initial use. An area of the Site will be designated for decontaminating equipment and materials. Decontamination residues will be managed and disposed of in accordance with relevant regulatory requirements.

5.12.1 Small Tools

Small tools and other apparatus used for sampling, such as trowels, spoons, corers, or borers, will be washed in a detergent and water solution (e.g., Alconox® or Liquinox®) and rinsed with tap water to remove particulates. Field filtration equipment (if required) will be rinsed with dilute nitric acid. The equipment will then be rinsed with methanol. The final step will be a distilled or deionized water rinse. Following decontamination, the equipment will be wrapped in aluminum foil to prevent possible contamination prior to the next use.

A similar decontamination protocol will be employed by the analytical laboratory when the fish are being prepared for fillet samples or homogenates.

5.12.2 Monitoring Equipment

Monitoring equipment, including water level sensors, pH probes, slugs, and pressure transducers, will be rinsed with distilled water and methanol between uses.

5.12.3 Investigation-Derived Waste

With the permission of CBS Corporation (CBS), the liquid investigation-derived waste (IDW) will be disposed of at the barrier well groundwater treatment plant located at the former Westinghouse Horseheads plant site. Characterization of any such liquid IDW will be in accordance with CBS directions prior to disposal.

Solid IDW from field sampling activities will be disposed of as commercial trash. Excess samples, including both abiotic and fish samples, will be disposed of by the laboratory in accordance with their SOPs and any applicable permit requirements.

6.0 SAMPLE DOCUMENTATION AND CUSTODY

This section describes the procedures to be used to identify samples, document sample collection, and maintain sample custody.

6.1 SAMPLE IDENTIFICATION

Cummings/Riter will use an identification numbering system to describe all samples collected during RI field activities. This number will identify the sample media and location. Samples from the various locations will have the following prefixes:

- Surface Water: SW08-,
- Sediment: SD08-,
- Potential Sources: PS08-, and
- Fish: CC08- (common carp); WS08- (white sucker); LB08- (largemouth bass); PS08- (pumpkinseed); BC08- (black crappie); GS08- (green sunfish); ** - (others depending on species collected).

The "08" designation indicates that the sample was collected in 2008 and differentiates these RI samples from prior samples that employed similar labeling. The identification will also include a number to allow for identifying the location from which the sample was collected or the sequential sample number (for fish). If more than one sample is collected at a specific location, the depth interval may also be used to modify the sample identification. For example, a sample identified as SD08-2 (6-18") indicates a sediment sample collected at Location SD-2 in 2008 from a depth of 6 to 18 inches.

6.2 SAMPLING DOCUMENTATION

Sampling personnel will document sampling activities on sample collection forms. The following information at each sample location will be recorded, as appropriate: the time the sample was collected, sampling personnel, sample number, specific conductance, temperature, water level, and any field observations.

Sample collection forms will be maintained in the project file in Cummings/Riter's Pittsburgh, Pennsylvania, office. After completion of the RI, the information will be transferred to a document repository established by the Group.

6.3 SAMPLE CUSTODY

The appropriate chain-of-custody for the samples collected during the RI field activities will be followed. Custody procedures are described in Section 5.0 of the QAPP (Appendix B).

REFERENCES

New York State Department of Environmental Conservation, January 1999. *Technical Guidance for Screening Contaminated Sediments*, Albany, New York.

New York State Department of Environmental Conservation, October 2002, *Draft Procedure for Collection and Preparation of Aquatic Biota for Contaminant Analysis,* Division of Fish, Wildlife and Marine Resources, Bureau of Habitat, Albany, New York.

U.S. Environmental Protection Agency, October 1988, *Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA*, OSWER Directive 9355.3-01, EPA/540/G-89/004, Office of Emergency and Remedial Response, Washington, D.C.

U.S. Environmental Protection Agency, December 1991. *Draft Analytical Method for the Determination of Acid Volatile Sulfide in Sediment,* EPA 821/R-91-100.

U.S. Environmental Protection Agency, February 1995. *Guidance for Risk Characterization.* Science Policy Council.

U.S. Environmental Protection Agency, December 1999. *Innovative Technology Verification Report – Sediment Sampling Technology.*

U.S. Environmental Protection Agency, November 2000. *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Third Edition.* Four volumes. Office of Water, Washington, D.C.

TABLES

TABLE A-1

SUMMARY OF SAMPLING PROGRAM

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS[a]	SAMPLES	FIELD DUPLICATES	MS/MSD SAMPLES	EQUIPMENT RINSATE BLANKS[b]	TRIP BLANKS[c]
Surface Water and Seeps	Oxidation/ Reduction Potential, pH, Temp., Specific Conductance, Dissolved Oxygen	Full TCL/TAL Plus ammonia, fluoride, hardness, nitrites, and total suspended solids	13[d]	1[d]	1[d]	1[d]	1[d]
Sediment[e]	pH, Oxidation/ Reduction Potential	Full TCL/TAL, and total organic carbon	17-44	2	2	2	2
Fish Tissue	--	TCL PCBs/Pesticides, TAL, lipid content[f]	12-24	--	1-2	--	--
Pipe Floc	--	Full TCL/TAL	1	--	--	--	--
Barrier Well Treated Discharge/ Cutler-Hammer Discharge/ Chemung Street Outfall	Oxidation/ Reduction Potential, pH, Temp., Specific Conductance, Dissolved Oxygen	Full TCL/TAL Plus ammonia, fluoride, hardness, nitrites, and total suspended solids	1 (each potential source)	--	--	--	--

(a) Parameters include: Full TCL/TAL includes VOCs, SVOCs, pesticides/PCBs, and TAL inorganics. TAL inorganics analyses of aqueous samples will include both the dissolved and total fractions.

(b) Equipment rinsate blanks will not be collected if disposable sampling tools are used.

(c) One trip blank will be shipped with each container submitted to the laboratory for VOC analyses. The total number of trip blanks in the table is an estimate.

(d) Samples to be analyzed sequentially. The numbers of actual QC samples will be prorated according to the actual number of field samples.

(e) Select sediment samples (up to six) will be analyzed for grain size analysis and for acid volatile sulfide/simultaneously extracted metals (AVS/SEM). The locations selected for grain-size and AVS/SEM analyses may or may not correspond.

(f) If upon field inspection collected fish specimens show deformities that may be indicative of PAH impacts, selected fish tissue samples will also be analyzed for TCL SVOCs.

TABLE A-2

SAMPLE CONTAINERS, VOLUMES, PRESERVATIVES, AND HOLDING TIMES

PARAMETER	CONTAINER	CONTAINER VOLUME	NO. OF CONTAINERS	PRESERVATIVES	HOLDING TIME [1]
Water:					
TCL VOCs	glass	40 ml septa	3	HCl	10 days
TCL SVOCs	glass	1000 ml	2	Ice	7 days to extract; 40 days to analyze extract
TCL Pesticides/PCBs	glass	1000 ml	3	Ice	7 days to extract; 40 days to analyze extract
TAL Inorganics (total)	plastic	500 ml	1	HNO_3	6 months except for Hg 26 days
TAL Inorganics (dissolved fraction)	plastic	500 ml	1	HNO_3 if field filtered Ice if lab filtered	6 months except for Hg 26 days
Cyanide	plastic	250 ml	1	NaOH	14 days
Hardness	plastic	500	1	HNO_3	28 days
Fluoride, Nitrites (expressed as N), TSS	plastic	1000 ml	1	Ice	48 hours (Nitrites)
Ammonia	plastic	250 ml	1	H_2SO_4	28 Days
Sediments:					
TCL VOCs	glass	4 oz.	3	Ice	10 days
TCL SVOCs, Pesticides/PCBs	glass	8 oz.	3	Ice	14 days to extract; 40 days to analyze extract
TAL Inorganics	glass	8 oz.	1	Ice	6 months except for Hg 26 days
Total Organic Carbon	glass	4 oz.	1	Ice	14 days
Acid Volatile Sulfides/ Simultaneously Extracted Metals	glass	4 oz.	3	Ice	14 days to extract; 28 days to analyze extract
Fish Tissue:					
TCL Pesticides/PCBs and Lipid Content[a] TAL Inorganics	plastic bag	Whole Fish (wrapped in hexane-rinsed aluminum foil)	1	Ice	14 days to extract from thaw; 40 days to analyze extract 6 months except for Hg 26 days
Floc:					
TCL VOCs	glass	4 oz.	3	Ice	10 days
TCL SVOCs, TCL Pesticides/PCBs	glass	8 oz.	3	Ice	5 days to extract; 40 days to analyze extract
TAL Inorganics	glass	8 oz.	1	Ice	6 months except for Hg 26 days

(a) If upon field inspection, collected fish specimens show deformities that may be indicative of PAH impacts, selected fish tissue samples will also be analyzed for TCL SVOCs.

FIGURES

Prepared For:
KOPPERS POND RI/FS GROUP

CUMMINGS RITER CONSULTANTS, INC.

FIGURE A-1
PROPOSED POND AND OUTLET CHANNEL SAMPLING LOCATIONS
KENTUCKY AVE. WELLFIELD SITE - OU4
HORSEHEADS, NEW YORK

FIGURE A-2
POTENTIAL ONGOING SOURCES INVESTIGATION
KENTUCKY AVE. WELLFIELD SITE - OU4
HORSEHEADS, NEW YORK

CUMMINGS RITER CONSULTANTS, INC.

Prepared For:
KOPPERS POND RI/FS GROUP

hheads-5a.mxd

ATTACHMENT A-1

STANDARD OPERATING PROCEDURES

CUMMINGS/RITER CONSULTANTS, INC.
STANDARD FIELD OPERATING PROCEDURES

DETERMINATION OF pH IN AQUEOUS SAMPLES

1.0 Scope and Application

1.1 This method is applicable to drinking, surface, ground, waste and saline waters, and domestic and industrial wastes.

1.2 This method is adapted from Methods 150.1 and 150.2 United States Environmental Protection Agency (EPA) 600-4-79-020 "Methods for Chemical Analysis of Water and Wastes," (Standard Methods, 18th Edition) and Method 9040B EPA SW-864 Rev. 2, 1/95 "Test Methods for Evaluating Solid Waste."

2.0 Method Summary

2.1 The pH of the sample is measured electrometrically using a pH meter equipped with a gel-filled glass combination electrode.

3.0 Interferences

3.1 Coatings of oily or particulate materials can impair electrode response and accuracy. Remove films by gently cleaning the electrode with alconox soap and a potable water solution, then rinsing thoroughly with deionized water. If the electrode response is insufficient, then clean with a diluted hydrochloric acid solution and recalibrate.

3.2 Temperature effects can be controlled by bringing the temperature of the samples to within ±2.0 degrees Centigrade of the certified buffer solutions used for calibration.

4.0 Apparatus

4.1 Horiba U-22 water quality meter with gel-filled electrode.

4.2 YSI 556 water quality meter with gel-filled electrode.

4.3 Any comparable water quality instrument with gel-filled electrode.

5.0 Reagents

5.1 Certified unexpired secondary standard buffer solutions, pH 7.00, pH 4.00, pH 10.00, standard units (commercially available).

5.2 Laboratory-grade deionized water.

5.3 Date of preparation or date of receipt and the date the container was opened should be placed on each standard container and along with the known value. This information shall also be placed on the "Standard Solution Log Sheet."

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6.0 Calibration Procedures

General Instructions - Each pH probe shall be cleaned after each use period and calibrated prior to usage using a minimum of two (2) pH buffer standards which bracket the anticipated values to be measured. Buffer aliquots shall not be used more than once; buffer solutions shall be dated at the time of initial use.

6.1 Horiba U-22 Water Quality Meter Calibration

6.1.1 Turn on the instrument using the power button; a low battery is indicated by E-1 (replace battery). Wash the sensor three times using deionized water. Place the instrument in the pH measurement mode with the Mode Key.

6.1.2 Zero Calibration (using pH 7.00 standard):

- Immerse the probe in pH 7.00 solution and after stabilization, press the CAL key twice while the instrument is in the pH measurement mode (the Man and Cal bars should be indicated). Use the up/down arrows to adjust the pH at the solution temperature.
- Press the ENT key: Manual calibration starts. When calibration is finished, the Data In light stops blinking and remains on. Press the CAL key to complete the calibration and move to the Manual Span calibration.

6.1.3 Span Calibration:

- After the zero calibration is complete and the unit is displaying the Man Span and CAL lights, place the probe in either pH 4.00 or pH 10.00 standard depending on what is expected in the samples to be tested.
- Use the up/down arrows to adjust the value to the correct one for the temperature of the solution. After stabilization, press the ENT key and the values flash until stable. When flashing is completed, and the Data In lights up, press the Meas key to return to measurement mode. The instrument is ready to measure pH values.

6.1.4 Auto Calibration:

- Wash the sensor three times with deionized water and place in the Auto Cal solution. (Fill the calibration breaker to the proper level as indicated).
- Press the CAL key in the pH, cond, turb, or D.O. measurements modes. The Auto and Cal bars will light up. When stable, press the ENT key to start Auto Cal. When pH has calibrated, it will stop blinking and remain on. Auto Cal will calibrate pH, cond, turb, D.O, and DEP. If an error message is displayed, see the

trouble shooting section of the instruction manual.
When all lights stop blinking, Auto Cal is complete.

- Press the Meas key to return to the measurement mode.
  The instrument is ready to measure pH values.

6.2 YSI 556 Water Quality Meter Calibration

6.2.1 Turn on instrument. Access calibrate screen by pressing the Escape key. Move bar to calibrate, hit Enter, then move bar to highlight pH; select 1-point, 2-point, or 3-point options. Press Enter for 3-point calibration, which provides the most accuracy when the expected value is unknown, or 2-point if the expected samples lie between 2 points.

6.2.2 Select the first pH Standard (use pH 7.0 first), or pour about 30 ml into calibration cup when upright.
Rinse sensor off with deionized water before immersion to avoid cross contamination.
Gently rotate and/or move probe up and down to remove trapped bubbles from the sensor. The pH sensor must be completely immersed for proper calibration.
Use the keypad to enter the calibration value and press Enter.
Allow at least one minute for stabilization, then if there is no significant change for 30 seconds, press Enter again. The screen will indicate if the calibration is accepted.
Press Enter again, and you will return to the calibration screen for the next standard to be calibrated.

6.2.3 Rinse probe, calibration cup, and screen in laboratory-grade deionized water. Pat dry.
Place next pH solution in calibration cup. Enter pH value on screen. Place probe in cup and press Enter. Allow at least one minute for equilibration. After 30 seconds with no significant change, press Enter. The screen will indicate if the calibration has been accepted. Press Enter and return to screen for the third standard value (if required) to be entered.

6.2.4 Repeat procedure as in 6.2.2 for third standard (if required).

6.2.5 Subsequent calibrations can be done with a 2-point calibration at pH 4.0 and 7.0 standard. Repeat 6.2.1 selection 2-point calibration and 6.2.2 using pH 4.0 and 7.0 standard.

7.0 Sample Analysis Procedure

7.1 Record sample temperature.

7.2 Analyze and record the pH and temperature of each sample using the temp probe and pH probe.

7.3 Rinse the electrode thoroughly with deionized water and blot dry between each sample.

7.4 Record results in the field data log (9.3).

8.0 Quality Control

8.1 Document the date, time, and the values obtained. If the difference between the expected value of the pH 10.00 check standard and the value measured is greater than ±0.10 pH units, recalibrate the meter with a two-point calibration at pH 4.0 and 7.0 to within ±0.05 pH units, as per Section 6.0 of this method.

8.2 Check calibration after 20 samples or every three hours with a 1-point calibration, as described in Sections 6.1.4 or 6.2.1. If check is not within ± 0.20 units, recalibrate per Section 6.0.

8.3 Dispose of all buffer solutions after expiration date.

9.0 Format for Calibration Log

9.1 YSI 556.

9.2 Horiba UV-22.

9.3 Field Data Logs.

9.4 Standard Receipt Log.

10.0 Management Approval

_____ _____

Approval Signature Date

_____

Title

CUMMINGS RITER

CUMMINGS/RITER CONSULTANTS, INC.
STANDARD FIELD OPERATING PROCEDURES

DISSOLVED OXYGEN

1.0 Scope and Application

1.1 This method is applicable to most wastewaters, surface waters, ground, and saline waters.

1.2 This Dissolved Oxygen (DO) probe technique gives comparable results on all sample types and is an excellent method in polluted and highly colored water, as well as strong waste effluents.

1.3 This method is adapted from United States Environmental Protection Agency (EPA) Method 360.1 (Standard Methods, 18th Edition).

2.0 Method Summary

2.1 The probes use Clark-type membrane-covered polarographic sensors with built-in thermistors for temperature measurement and compensation. A thin permeable membrane stretched over the sensor isolates the sensor elements from the environment and allows oxygen and certain other gases to enter.

2.2 A polarizing voltage is applied across the sensor; oxygen that has passed through the membrane reacts at the cathode, causing a current to flow.

2.3 The membrane passes oxygen at a rate proportional to the pressure difference across it. Since oxygen is rapidly consumed at the cathode, it can be assumed that the oxygen pressure inside the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the absolute pressure of oxygen outside the membrane. If the oxygen pressure increases, more oxygen diffuses through the membrane and more current flows through the sensor. A lower pressure results in less current.

3.0 Interferences

3.1 This method is not subjected to the serious errors of other methods caused by interferences such as oxidizing, reducing agents, nitrate ion, ferrous iron, and organic matter. The sensing element is protected by an oxygen-permeable plastic membrane that serves as a diffusion barrier against impurities.

3.2 Halogens, Neon, Nitrous Oxide, and CO are interferences. If erroneous readings are suspected, it may be necessary to determine if these interference gases are the cause.

3.3 The Winkler Azide Modified Method is subject to additional interference, such as ferrous iron or ferric iron in high concentrations; other oxidizing or reducing substances other than nitrates, even high suspended solids (see Standard Methods: Oxygen [dissolved] for Alternative Methods).

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4.0 Apparatus
- 4.1 Horiba U-22.
- 4.2 YSI 556 Water Quality Meters.
- 4.3 Calibration Chambers.
- 4.4 BOD Bottle, Erlenmeyer Flask.
- 4.5 Buret 25-milliliter (ml) Class "A" and 250-ml graduated cylinder "Class A."

5.0 Reagents
- 5.1 Iodate-Iodide Standard Solution (0.0125 N).
- 5.2 DO Zero Calibration Solution.
- 5.3 Sodium Sulfate (Na2S03) Granular Solid.
- 5.4 Cobalt Chloride (CoC12) Granular Solid.
- 5.5 Manganous Sulfate Granular Solid or Liquid.
- 5.6 Alkaline Iodide-Azide Reagent.
- 5.7 Sulfamic Acid Powder.
- 5.8 Sodium Thiosulfate 0.025 N Solution.
- 5.9 Starch Indicator Solution.
- 5.10 Date of preparation or date of receipt and the date the container was opened should be placed on each standard container and along with the known value. This information shall also be placed on the "Standard Solution Log Sheet."

6.0 Calibration Procedures
- 6.1 Check probe membrane if bubbles or damage are apparent.
  - 6.1.1 Replace membrane if erratic readings are observed or calibration is not stable. The anode may become contaminated with biological or silver chloride coating, which must be removed. See cleaning procedures in the instrument-specific manual.
- 6.2 Prepare Instrument (set instrument in its operating position; vertical or on its back). Turn on instrument.
  - 6.2.1 Each instrument manual or pocket instruction card will describe the correct sequence for pressing the proper keys.
  - 6.2.2 For the YSI 556 and Horiba U-22, you will need to know the current barometric pressure for your location. Also, the salinity of the standard will need to be known (usually 0.0 parts per thousand [ppt]).
- 6.3 Zero Calibration/Check
  - 6.3.1 Use a solution of sodium sulfite and cobalt chloride dissolved in tap water or deionized water, or a premixed packet of zero DO solution (manufactured by Calitech) can be used.
  - 6.3.2 Add about 50 gm of sodium sulfite and 0.5 gm of cobalt chloride to one liter of deionized or tap water if using this method. Stir the mixture to dissolve.

6.3.3 Wash probe with deionized water and place in zero solution. Allow time for stabilization. Readings should be between 0.0 and 0.1 parts per million (ppm). Record or check results.

6.4 Saturated Air Calibration

6.4.1 For the YSI 556, saturated air calibration is conducted daily before sampling begins and again every three hours of operation.

6.4.2 Place the pre-cleaned probe being standardized into its specific calibration chamber with a moist sponge or 1/8 inch of water (see specific instrument manual). Allow several minutes for the readings to stabilize. When stable, proceed as prompted by the manual.

6.4.3 Determine the altitude and/or local barometric pressure to enter at this point.

6.4.4 Enter salinity (usually 0.0 ppt).

6.4.5 Continue to read in percent saturation or milligrams per kilogram (mg/kg), record results in ppm, and temperature in degrees (°) C.

6.4.6 If this is a daily calibration, proceed to sample measurement.

6.4.7 If this calibration is done prior to the weekly Winkler titration for a calibration check, proceed to the Saturated Water Calibration Method.

6.5 Saturated Water Calibration Method

6.5.1 This method can be used as a secondary method for the YSI 556 and Horiba U-22 for calibration, but usually cannot be conducted in the field. It is used as a secondary check in the laboratory in conjunction with the Winkler Method.

6.5.2 Air saturate a volume of water by aerator for at least 15 minutes at a relatively constant temperature.

6.5.3 Place the probe in the sample and stir. Refer to Air-Saturated water tables provided in the instruction manual at that temperature for the corresponding value in mg/l.

6.5.4 Record Value. At this point, collect a sample in a BOD bottle, cap off, allowing no air bubbles. Proceed to the Winkler Method for the actual value to the air-saturated water.

6.6 Winkler Buret Titration Method of Standardization: This method is conducted every seven days. It is a check against the other methods of calibration.

6.6.1 Collect a water sample from a common source in a clear glass-stoppered BOD bottle.

Note: Overflow the bottle for two to three minutes to remove any trapped air bubbles.

6.6.2 Add the proper amount (eight drops) of Manganous Sulfate and Alkaline Iodide-Azide Solution Reagent.

Note: Reagents used for the Winkler Method are part of the LaMotte Dissolved Oxygen Kit, Model EDO.

6.6.3 Immediately insert the stopper so that no air is trapped in the bottle. Invert several times to mix.
Note: A precipitate will form. It will be orange-brown if oxygen is present or white if oxygen is absent.

6.6.4 Wait until the floc in the solution has settled. Again invert the bottle several times and wait until the floc has settled.
Note: Results will not be affected if the floc does not completely settle.

6.6.5 Remove the stopper and add the required amount (1.0 gram) of Sulfamic Acid Powder. Replace the stopper without trapping air in the bottle, and invert several times to mix until totally dissolved. (This is the prepared sample.)

6.6.6 Pour the prepared sample into a 25-ml graduated cylinder to the 20-ml mark.

6.6.7 Fill the titrator syringe with 0.025 N Sodium Thiosulfate Solution to the zero mark, and titrate the prepared sample with the Sodium Thiosulfate Solution to a pale yellow color.

6.6.8 Add eight drops of starch solution and swirl to mix. A dark-blue color will develop. Continue the titration with the syringe until the solution changes from dark blue to colorless. See note for interferences.

6.6.9 Record the test result from the scale on the syringe. Units are in ppm.

7.0 Sample Analysis Procedure

7.1 With the instrument prepared for use and the probe calibrated, place the probe in the sample to be measured and provide stirring, or in a flow cell, no stirring is required.

7.2 If the submersible stirrer is not used, provide manual stirring by raising and lowering the probe about 1 foot per second.

7.3 Allow sufficient time for probe to stabilize to sample temperature and DO. Read and record the DO in mg/l or ppm; also record temperature in °C.

8.0 Sources of Error

8.1 If there are bubbles under the membrane or wrinkled membrane, replace membrane as needed.

8.2 If the cathode becomes tarnished (it is not bright), recondition probe or send it in for factory service.

8.3 If the anode is silver, the anode may become contaminated, which will prevent successful calibration. Soak overnight in 3 percent ammonia solution or physically remove tarnish with 400 grit wet/dry sand paper.

9.0 Quality Control

9.1 Accuracy Check: Recalibrate at the beginning of the sample run and every three hours. Also, if system appears to be erratic, recalibrate before each sample by the zero DO or the Saturated Air Method.

9.1.1 A sample with zero DO should be prepared when analyzing samples with low DO. (Add excess solution sulfite, Na2S03, and a trace of Cobalt Chloride, CoC12, to bring DO to zero or a premixed zero DO solution zero.) Zero DO solution manufactured by Calitech can also be used.

9.2 Precision Check: Run 10 percent of samples in duplicate, if possible; duplicates are not necessary if using a flow cell.

9.3 Calibrate the DO meters against the Winkler Method at least once per week or prior to use if sample period is no longer than one week.

9.3.1 Draw a volume of water. Determine the oxygen value by the Winkler titration technique.

9.3.2 Place the probe in a second sample and stir.

9.3.3 Set the salinity and barometric pressure.

9.3.4 Adjust the value to that found by Winkler titration. Allow the probe to remain in the sample for at least two (2) minutes before setting the calibration value and for two (2) more minutes to verify stability. Readjust if necessary.

9.4 Accuracy check of Winkler Standard Solution

9.4.1 Add the required amount of Sulfamic Acid Powder to 200 ml Iodate-Iodide Standard Solution (0.00125 N).

9.4.2 Titrate this solution with 0.025 N Sodium Thiosulfate and follow Sections 6.6.2 through 6.6.9.

9.4.3 This check solution is equivalent to 10 mg/l DO. Thus, the volume if the titrant used should be 10 ml. If more than 10.5 ml is required to reach the endpoint, discard the Sodium Thiosulfate solution and acquire a fresh batch; recheck new solutions.

9.4.4 Run this check every six months or more often as expiration of the Sodium Thiosulfate approaches.

10.0 Format for Calibration and Sign Off Logs

YSI 556.
Horiba U-22.
Field Data Logs.
Winkler Calibration Log.
Winkler Titration Log
Winkler Standards Log.
Winkler Accuracy Check Log.

11.0 Management Approval

Approval Signature	Date
Title	

CUMMINGS RITER

CUMMINGS/RITER CONSULTANTS, INC.
STANDARD FIELD OPERATING PROCEDURES

DETERMINATION OF SPECIFIC CONDUCTIVITY IN AQUEOUS SAMPLES

1.0 Scope and Application

1.1 This method is applicable for the determination of specific conductance in surface, drinking, ground and saline waters, also domestic and industrial wastes.

1.2 This method is adapted from United States Environmental Protection Agency (EPA) Method 120.1 (Standard Methods, 18th Edition).

2.0 Method Summary

2.1 Specific Conductance is measured using a Horiba U-22 or YSI 556 conductance meter equipped with a 1.0 centimeter (cm) conductivity cell. Results are reported as ohm/cm at 25 degrees C (°C).

2.2 It is preferable that samples be analyzed near 25°C. This is usually not practical. With the Horiba U-22 and YSI 556 temperature compensated conductivity is automatically corrected to 25°C with a temperature coefficient of 2 percent per °C.

3.0 Reagents

3.1 Purchased certified standards can also be used with various standards prepared from stock standards (initial 5 point standard curve will be prepared from 71.8 mS/m, 0.667 S/m, and 5.87 S/m standards).

3.2 Laboratory-grade deionized water.

3.3 Conductivity cell-cleaning solution – prepared 100 ml of 10 N HCl by carefully adding 83 ml of concentrated HCl to 17 ml of deionizer water. Mix the 10 N HCl 1:1 with Isopropyl Alcohol. Clean the electrode in the cleaning solution for two minutes. Rinse thoroughly with deionized water. Air dry before use. Also soaking the conductivity cell in Simple Green detergent at full strength can be used.

3.4 Date of preparation or date of receipt and the date the container was opened should be placed on each standard container and along with the known value. This information shall also be placed on the "Standard Solution Log Sheet."

4.0 Apparatus

4.1 Horiba U-22 water quality meter.

4.2 YSI 556 water quality meter.

4.3 Any comparable water quality meter.

4.4 Rinsing flasks.

4.5 NIST traceable thermometer.

CUMMINGS RITER

5.0 Calibration of Meters

5.1 Each meter must be calibrated prior to daily activities using quality control (QC) standards that have been either prepared or purchased. Standards will be selected depending on the meter ranges and values to be expected in the samples (see the specific meter's calibration procedures for those ranges).

5.1.1 Each meter should be zeroed according to equipment instructions before calibration. If there are no specific instructions to enter into the meter, check the probe in deionized water of 8 uS/cm or less. Record value or check off that this has been done.

5.1.2 Calculate the results and the percent recovery for the standard. If the results are not within 10 percent of the known value, re-analyze an additional aliquot of the standard. If this value is not within specifications, obtain a fresh calibration standard and analyze. If values fail to come within specifications, clean or replace the conductivity probe and recalibrate. Also, check batteries in meter.

5.1.3 After calibration, recheck one standard in the expected range and record value. Do not readjust unless the whole calibration procedure is to be repeated.

5.1.4 Continuing calibration is run for each meter after three hours of continuous use or every 20 samples, whichever is less. (See procedure for each meter.) One standard solution will be selected to recheck calibration, and the result noted or checked if within 10 percent of expected value.

5.1.5 A QC check sample is to be run every 20 samples (see QA Section). Its value is to be different from normal standards, but near the range of samples being tested.

5.1.6 All meters used by Cummings/Riter Consultants, Inc. automatically compensate for temperature variations.

5.2 The Horiba U-22

The Horiba U-22 must be calibrated in the span mode with three conductivity standards: one in each range (0.0 – 99.9 mS/m, 0.090 – 0.999 S/m; and 0.90 – 9.99 S/m) after the unit has been zeroed in air (see 5.2.2).

5.2.1 Potassium chloride standards to be selected from for each range:

KCL Standard	Conductivity	Range to be Calibrated
0.005 mol/L	71.8 mS/m	0.0 to 99.9 mS/m
0.050 mol/L	0.667 S/m	0.090 to 0.000 S/m
0.500 mol/L	5.87 S/m	0.90 to 9.99 S/m

5.2.2 Zero Calibration: Wash probe three times with laboratory-grade deionized water; shake off, and pat dry. Press CAL key twice in Conductivity mode. Use up or down keys to set the value to 0.0. Press Enter key. DATA IN light blinks until value has stabilized. DATA IN light stays on when zero calibration is complete.

5.2.3 Span Calibration: Wash probe two to three times with laboratory-grade deionized water and shake off. Fill calibration cup with appropriate amount of standard solution and immerse sensor in it. Press CAL key to make sure instrument is in Span Calibration mode. Use up or down keys to set standard solution value. Press Enter key. DATA IN light blinks until indicated value stabilizes. DATA IN light stays on when the calibration is complete. Repeat for all three ranges.

5.3 YSI 556

The YSI 556 is standardized daily by a two-point calibration within the expected range of sample readings. If a sample is out of range by a factor of 10 from the standard, recalibrate at the new range. A zero check must be accomplished before the standard is run (see 5.3.2).

5.3.1 Potassium Chloride Standard:

Conductivity um/cm	Range to be Calibrated
0.667 S/m	0 - 1 S/m
5.87 S/m	1 - 105 S/m

5.3.2 Zero Check: Place the probe in deionized water with a known value and observe results. Clean probe or replace if reading does not stabilize or read at or near expected value. Remove probe, pat dry, and observe reading in air. It should be 0.0000.

5.3.3 Calibration of the YSI 556 with the unit on:

- Press the Escape key to display the main menu.
- Use the arrow keys to highlight Calibrate.
- Press Enter.
- Use the arrow keys to highlight Conductivity.
- Press Enter.
- Place enough calibration standard into a clean, dry pre-rinsed calibration cup to cover probe. Note: For maximum accuracy the conductivity standard you choose should be within the same conductivity range as the samples are expected to be. (See 5.3.1)
- Immerse the sensor into the solution.
- Gently rotate or move the probe module up and down to remove air bubbles from the cell.

- Use the keypad to enter the value of the standard you are using in units of mS/cm @ 25°C.
- Press Enter. Allow at least one minute for temperature equilibration. Observe the reading under conductivity. When the reading shows no significant change for 30 seconds, press Enter.
- The screen will indicate that the calibration has been accepted. Press Enter to continue.
- Press Enter again to return to the Conductivity Calibrate screen.
- Press Escape to return to the Calibrate menu.
- Press Escape again to return to the Main menu.

5.3.4 Recheck with a conductivity calibration solution other than 1,409 um/cm. Record the results. If out by +/- 10 percent, replace probe.

6.0 Procedure for Sample Analysis

6.1 Allow samples and standards to temperature equilibrate prior to analysis (which is not always possible in the field).

6.2 Set up a deionized water squirt bottle and paper towels with extra rinsing flasks nearby.

6.3 Mix the sample thoroughly prior to analysis if a grab sample, or take a reading if in a flow cell. No mixing is necessary when using a flow cell.

6.4 Dispense an aliquot of the sample into a clean polyethylene container.

6.5 Rinse the thermometer thoroughly with deionized water. Blot dry with a clean paper towel. Measure and record the temperature of the sample in degrees C (most meters have a temperature readout already, and therefore, this step may not be necessary).

6.6 Rinse the conductivity cell thoroughly in deionized water. Blot dry.

6.7 Immerse the cell into the sample several times, expelling all air through the vent. Allow the solution to stabilize.

6.8 All samples are analyzed as required by work plan if using a flow cell.

6.9 Rinse both the thermometer (if required to use one) and the conductivity cell thoroughly between samples.

6.10 Record temperature in degrees C and conductivity results in the field log (see Section 9.4).

7.0 Quality Control

7.1 All samples are analyzed according to the work plan.

7.2 A QC check sample of 7,000 umho/cm at 25°C, or similar value standard, not usually run for routine standardization will be analyzed every 20 samples and value recorded on the calibration log for that day.

7.3 Percent recovery where:

$$\% \text{ recovery} = \frac{\text{observed value um/cm @ 25°C}}{\text{100 X STD value or check STD um/cm}}$$

If the results are not within 10 percent of the known value, reanalyze or prepare a new aliquot; clean; or replace probe.

7.4 Check prepared standards and purchased standards every three months against a benchtop conductivity meter with a platinum probe. If percent recovery is out by more than 10 percent, discard the standard.

8.0 Format for Calibration Log

8.1 If the standards and check standards are acceptable, report the results in whole numbers as follows:

Conductivity	Report to nearest
71.8 mS/m	1
0.667 S/m	1
5.87 S/m	1

9.0 Format of Calibration Signoff Log and Field Log

9.1 YSI 556.

9.2 Horiba U-22.

9.3 Field Log.

9.4 Standard Preparation/Receipt Log.

10.0 Management Approval

_____ _____

Approval Signature Date

_____

Title

CUMMINGS/RITER CONSULTANTS, INC.
STANDARD FIELD OPERATING PROCEDURES

DETERMINATION OF TEMPERATURE IN AQUEOUS SAMPLES

1.0 Scope and Application
- 1.1 This method is applicable to drinking, surface and waste waters, and domestic and industrial wastes.
- 1.2 This method is adapted from United States Environmental Protection Agency (EPA) Methods 170.1 and SM 2550B (20th Edition, 1998).

2.0 Method Summary
- 2.1 The temperature of the sample is determined by measuring the average kinetic energy of the particles in a sample of water, expressed in terms of degrees designated on a standard scale.

3.0 Interferences
- 3.1 Water temperature that is not homogeneous due to stratification.

4.0 Apparatus
- 4.1 Horiba U-22 water quality meter.
- 4.2 YSI 556 water quality meter.
- 4.3 ERTCO NIST traceable thermometer.
- 4.4 Any comparable water quality instrument capable of measuring temperature.

5.0 Calibration Procedures

General Instructions - The meters shall be calibrated quarterly, or as needed, by comparing the reading of the NIST traceable thermometer with that of the water quality instrument. The calibration may be difficult to perform in the field due to weather and other environmental interferences; therefore, the temperature calibration may be done prior to field activities.

6.0 Horiba U-22
- 6.1 Place the required amount of water (tap water is sufficient) in the calibration beaker to submerge the water quality meter temperature probe. Stir the water in the beaker to make sure the temperature is homogeneous. Place the ERTCO NIST thermometer in the water, and allow the thermometer to equilibrate for several minutes.
- 6.2 After the temperature reading has stabilized, note the reading (in degrees Celsius [°C]) and immediately place the water quality meter in the beaker.
- 6.3 Make sure the meter is in temperature measurement mode and allow the meter to stabilize for several minutes. Note the temperature reading after stabilization. If the temperature difference between the NIST traceable thermometer and the Horiba U-22 is 1°C or less, then meter calibration is

not needed. If the difference is greater than 1°C, proceed with the temperature calibration procedures.

6.4 Leave the water quality meter probe submerged in the water for calibration. Press the CAL key in the temperature measurement mode. Use the up or down keys to set the temperature to equal the temperature measured using the NIST traceable thermometer.

6.5 Press the Enter key and the DATA IN light will blink. When the value stabilizes, the DATA IN light will stop blinking and the calibration is complete.

7.0 YSI 556

7.1 There are no specific instructions to enter into the meter for a temperature calibration; therefore, a check of the probe with the NIST traceable thermometer is required, as follows:

- Put the instrument in the temperature measurement mode.
- Place enough water into a clean, dry pre-rinsed calibration beaker to cover probe. Immerse the NIST traceable thermometer into the water and allow it to equilibrate.
- Allow at least one minute for temperature equilibration. Observe the temperature reading. When the reading shows no significant change for at least one minute, the temperature has stabilized.
- Record value of measurement. Next, immediately insert the probe into the water with the known temperature, and allow the reading to stabilize. Verify that the measurement is within ten percent of the anticipated value, and check off that this has been done on the calibration form. If the measurement is not within ten percent, discard the water and retry the procedure with new water.

8.0 Sample Procedures

8.1 With the instrument prepared and calibrated (if needed), place the probe in the flow cell or container.

8.2 Allow sufficient time for the probe to stabilize to sample temperature. Read and record the temperature in °C.

9.0 Quality Control

9.1 Document the date, time, and the values obtained.

9.2 Check calibration quarterly, as described in Sections 5.0 and 6.0.

10.0 Sources of Error

10.1 Flow cell or container where probe is placed should be kept out of direct sunlight, if possible, to prohibit water from being heated.

11.0 Format for Calibration Log

11.1 Horiba U-22.

11.2 YSI 556.
11.3 Calibration Data Logs.

12.0 Management Approval

_____ _____
Approval Signature Date

_____
Title

CUMMINGS RITER

CUMMINGS/RITER CONSULTANTS, INC.
STANDARD FIELD OPERATING PROCEDURES

DETERMINATION OF OXIDATION-REDUCTION POTENTIAL (ORP) IN AQUEOUS SAMPLES

1.0 Scope and Application

1.1 This method is applicable for the determination of Oxidation Reduction Potential (ORP) in surface, drinking, ground, and saline waters, also domestic and industrial wastes. ORP is the potential at which oxidation occurs at the anode (positive) and reduction occurs at the cathode (reduction) of an electrochemical cell.

1.2 This method is adapted from Standard Method 2580B (Standard Methods, 20th Edition, 1998).

2.0 Method Summary

2.1 ORP is measured using a Horiba U-22 or YSI 556 which utilizing the platinum electrode method. Results are reported in millivolts (mV).

3.0 Reagents

3.1 YSI Zobell solution can be prepared by adding 125 milliliters (ml) of deionized water to purchased bottle containing correct amounts of potassium chloride, potassium ferrocyanide trihydrate, and potassium ferrocyanide.

3.2 Laboratory-grade deionized water.

3.3 Date of preparation or date of receipt and the date the container was opened should be placed on each standard container and along with the known value. This information shall also be placed on the "Standard Solution Log Sheet."

4.0 Apparatus

4.1 Horiba U-22 water quality meters.

4.2 YSI 556 water quality meter.

4.3 Any comparable water quality meter.

5.0 Calibration of Meters

5.1 Each meter must be calibrated prior to daily activities using Zobell solution that has been prepared and is unexpired. The calibration will be performed on an as-needed basis in accordance with the manufacturer's recommendations.

5.1.1 Each meter should be calibrated according to equipment instructions before calibration. If there are no specific instructions to enter into the meter, check the probe in the Zobell solution. Record value of measurement, verify that the measurement is

CUMMINGS RITER

within10 percent of the anticipated value, and check off this has been done on the calibration form.

5.1.2 If the results are not within 10 percent of the known value, re-analyze an additional aliquot of the standard. If this value is not within specifications, obtain a fresh calibration standard and analyze. If values fail to come within specifications, clean the probe and check again. Also, check the batteries in the meter and replace, as needed.

5.1.3 Continuing calibration is run for each meter after three hours of continuous use or every 20 samples, whichever is less.

5.2 The Horiba U-22

5.2.1 There are no specific instructions to enter into the meter for an ORP calibration, therefore a check of the probe in the Zobell solution is required, as follows:

- Put the instrument in the ORP measurement mode.
- Place enough calibration standard into a clean, dry pre-rinsed calibration beaker to cover probe. Immerse the sensor into the solution.
- Allow at least one minute for temperature equilibration. Observe the reading under ORP. When the reading shows no significant change for at least one minute, the probe has stabilized.
- Record value of measurement, verify that the measurement is within10 percent of the anticipated value, and check off this has been done on the calibration form. If the measurement is not within 10 percent discard solution, discard the solution, and retry with new solution.

5.3 YSI 556

5.3.1 The YSI 556 is standardized daily by a one point with the Zobell solution.

5.3.2 Calibration of the YSI 556 unit:

Press the Escape key to display the main menu.

- Use the arrow keys to highlight Calibrate.
- Press Enter.
- Use the arrow keys to highlight ORP.
- Press Enter.
- Place enough calibration standard into a clean, dry, pre-rinsed calibration cup to cover probe. Immerse the sensor into the solution.
- Gently rotate or move the probe module up and down to remove air bubbles from the cell. Screw the calibration cup onto the probe.

- Use the keypad to enter the value (mV) of the calibration solution you are using at the current temperature. The Zobell solution values are provided with the standard bottle.
- Press Enter. Allow at least one minute for temperature equilibration. Observe the reading under ORP. When the reading shows no significant change for 30 seconds, press Enter.
- The screen will indicate that the calibration has been accepted. Press Enter to continue.
- Press Enter again to return to the ORP Calibration screen.
- Press Escape to return to the Calibrate menu.
- Press Escape again to return to the Main menu.

6.0 Procedure for Sample Analysis

6.1 Allow samples and standards to temperature equilibrate prior to analysis (which is not always possible in the field, depending on ambient temperature).

6.2 Set up a deionized water squirt bottle and paper towels with extra rinsing flasks nearby.

6.3 Mix the sample thoroughly prior to analysis if a grab sample, or take a reading if in a flow cell if low flow techniques are utilized.

6.4 Dispense an aliquot of the sample into a clean polyethylene container if a flow cell is not used.

6.5 Immerse the cell into the sample and allow the solution to stabilize.

6.6 All samples are analyzed as required by an approved work plan if using a flow cell.

6.7 Rinse probe thoroughly between samples.

6.8 Record the ORP results on the correct form.

7.0 Quality Control

7.1 All samples are analyzed according to the work plan for the specific site.

7.2 A quality control check using the Zobell solution will be analyzed every 20 samples or every three hours, whichever comes first, and record the value on the calibration log for that day.

7.3 Check prepared standards every three months. If percent recovery is out by more than 10 percent, discard the standard.

8.0 Format for Calibration Log

8.1 If the standards and check standards are alright, report the results as whole numbers on the appropriate log.

9.0 Format of Calibration Signoff Log and Field Log
- 9.1 YSI - 556.
- 9.2 Horiba U-22.
- 9.3 Field Log.
- 9.4 Standard Preparation/Receipt Log.

10.0 Management Approval

_____ _____

Approval Signature Date

_____

Title

CALIBRATION SIGN OFF LOG

SITE_____ DATE_____

ANALYST_____ METER MAKE/MODEL

_____

Notes:

_____

☐ **pH Calibration**

Recheck pH 4.00 ± 0.20

Y N Cal (pH 7.00) ± 0.05 Value = _____

Y N pH 4.00 ± 0.05 **Quality Control Sample**

Y N pH 10.00 ± 0.10 Field value =_____ Actual Value=_____

☐ **Sp Conductance**

Y N Zero-check Recheck 1,409 mS/cm

Y N 718 mS/cm STD ±5% Value = _____

Y N 1,409 mS/cm STD ±5% **Quality Contol Sample**

Y N 2,764 mS/cm ±5% Field value =_____ Actual Value=_____

Y N 6,670 mS/cm ±5%

Y N 58,700 mS/cm ±5%

☐ **D.O. Calibration**

Date of Last Winkler STD _____ **Current Barometric pressure** _____

o/o Saturation **Current Temperature** _____

Y N Calibration accepted **Result** _____

☐ **Continuing Calibration** _____

Y N pH 7.0 Value = _____

Sp. Cond. 1,409 mS/cm Value = _____

D.O. Value =_____ Temp°C_____

☐ **Continuing Calibration** _____

Y N pH 7.0 Value = _____

Sp. Cond. 1,409 mS/cm Value = _____

D.O. Value =_____ Temp°C_____

NOTES/COMMENTS:_____

_____

_____

_____

_____

Analyst Signature _____

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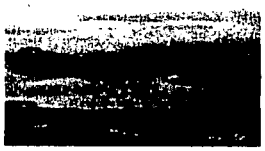
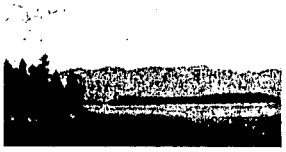
Aquatic Research Instruments

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Gravity Corer
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Core Extruding Apparatus
Discrete Point Piston Corer

PLANKTON SAMPLERS
Plankton Traps
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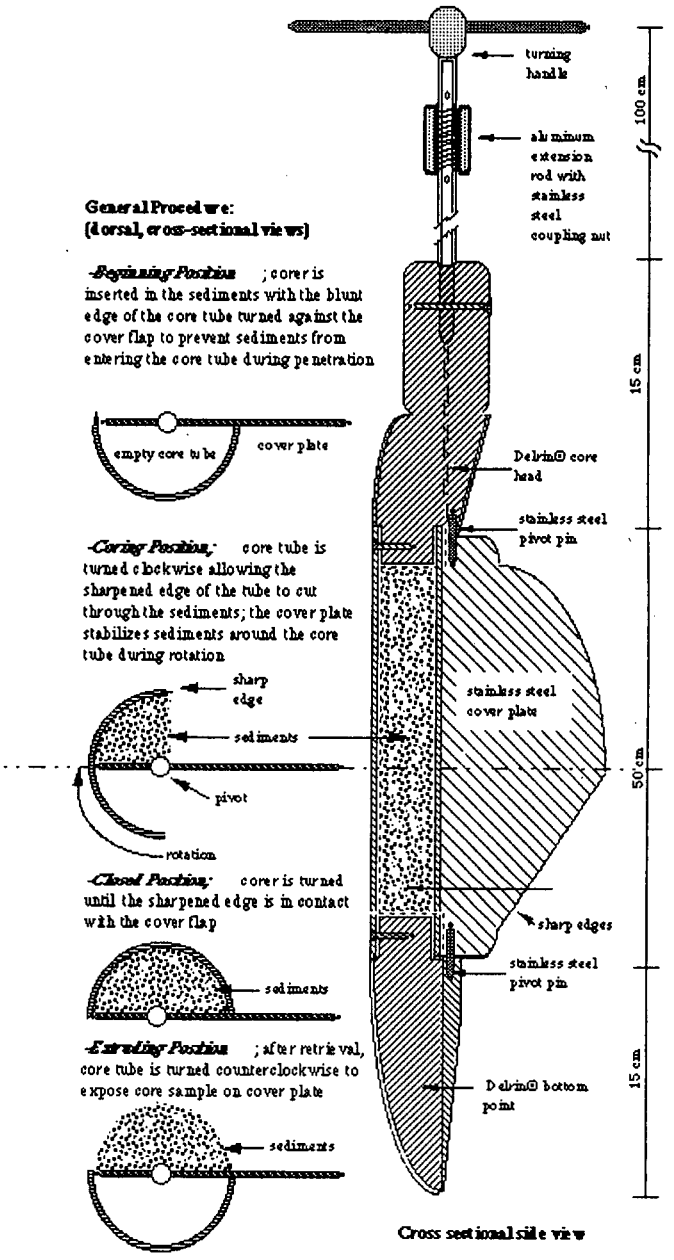
LABORATORY EQUIPMENT
Utermoehl (Phytoplankton) Sedimentation Chambers
Folsom Plankton Sample Splitter
Motoda Plankton Sample Splitter
Zooplankton Counting Trays

Russian Peat Borer



Paleoecological analysis of bog and salt marsh sediments

- Collection of uncompressed cores in poorly decomposed woody peat
- Shallow water applications

Russian Peat Borer Unit Includes:

- **stainless steel peat borer**
  - (50.0 cm X 5.4 cm O.D., 2.0 mm wall thickness)
  - one wall of the core tube is sharpened to longitudinally cut through sediments when sampler is turned clockwise
  - solid Delrin(r) core head and bottom point support a stainless steel cover plate which freely rotates inside the core tube
  - the stainless steel cover plate (2.0 mm thick) is curved and sharpened to minimize disturbance when inserted into the sediments
- **two aluminum extension rods**
  - (1.0 m X 1.9 cm diam.) with stainless steel coupling nuts
- **aluminum turning handle**
- **plastic carrying case**

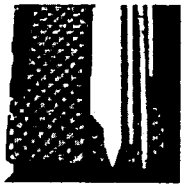
Notes: The Russian Peat Borer (7.5 cm x 60cm model shown) is a side filling chambered-type sampler. This discrete point sampler enables one to drive the sampler to any point in the sediment profile in the closed (empty) position.

Once the target depth is reached, the "T" handle is turned clockwise to initiate the sampling while the pivotal cover plate supports the cutting action of the bore. As the sampler is turned 180 degrees, the sharpened edge of the bore longitudinally cuts a semi-cylindrical shaped sample until the opposite side of the cover plate is contacted.

The contained sample can now be recovered without risk of contamination by overlying sediments. The sample is extruded from the bore by a counterclockwise rotation where sample rests on cover plate ready for sectioning.

STANDARD RUSSIAN PEAT BORER

- Stainless steel and delrin construction
- 5.0 cm x 50 cm diam.



- turning "T" handle
- 2-ea. Aluminum holobar extension rod w/ stainless steel couplers (40mm diam. x 1.2m)
- carry case
- **complete: $725**

- Aluminum holobar extension rod w/ stainless steel couplers (40mm diam. x 2.4m): **$122**
- Aluminum holobar extension rod w/ stainless steel couplers (40mm diam. x 1.2m): **$69**
- Slide hammer assembly, bronze and stainless steel construction: **$105**

Citations

Faegri, K., and J. Jvesen. 1975. Textbook of Pollen Analysis. Third edition, Hafner, N.Y., N.Y. Urso, S.B, S.W. Nixon, J.K. Cochran, D.J. Hirschberg, and C. Hunt, 1989. Accretion rates and sediment accumulation in Rhode Island salt marshes. Estuaries 12(4) 300-317. Jowsey, P.C., 1966. An improved peat sampler. New Phytologist 65: 245-248.

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United States
Environmental Protection
Agency

Office of Research and
Development
Washington DC 20460

EPA/600-R-01/009
December 1999

EPA

Innovative Technology Verification Report

Sediment Sampling Technology

Aquatic Research Instruments Russian Peat Borer

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, DC 20460

Environmental Technology Verification Program
Verification Statement

TECHNOLOGY TYPE: **Sediment Sampler**

APPLICATION: **CORE SAMPLING OF SEDIMENT**

TECHNOLOGY NAME: **AQUATIC RESEARCH INSTRUMENTS RUSSIAN PEAT BORER**

COMPANY: **AQUATIC RESEARCH INSTRUMENTS**
ADDRESS: **1 HAYDEN CREEK ROAD**
LEMHI, IDAHO 83466

WEB SITE: **http://www.aquaticresearch.com**

TELEPHONE: **(208) 756-8433**

VERIFICATION PROGRAM DESCRIPTION

The U.S. Environmental Protection Agency (EPA) created the Superfund Innovative Technology Evaluation (SITE) and Environmental Technology Verification (ETV) Programs to facilitate deployment of innovative technologies through performance verification and information dissemination. The goal of these programs is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. These programs assist and inform those involved in design, distribution, permitting, and purchase of environmental technologies. This document summarizes results of a demonstration of the Russian Peat Borer designed and fabricated by Aquatic Research Instruments.

PROGRAM OPERATION

Under the SITE and ETV Programs, with the full participation of the technology developers, the EPA evaluates and documents the performance of innovative technologies by developing demonstration plans, conducting field tests, collecting and analyzing demonstration data, and preparing reports. The technologies are evaluated under rigorous quality assurance (QA) protocols to produce well-documented data of known quality. The EPA National Exposure Research Laboratory, which demonstrates field sampling, monitoring, and measurement technologies, selected Tetra Tech EM Inc. as the verification organization to assist in field testing two sediment sampling technologies. This demonstration was funded by the SITE Program.

DEMONSTRATION DESCRIPTION

In April and May 1999, the EPA conducted a field demonstration of the Russian Peat Borer along with one other sediment sampler. This verification statement focuses on the Russian Peat Borer; a similar statement has been prepared for the other sampler. The performance and cost of the Russian Peat Borer were compared to those of two conventional samplers (the Hand Corer and Vibrocorer), which were used as reference samplers. To verify a wide range of performance attributes, the Russian Peat Borer demonstration had both primary and secondary objectives. Primary objectives for this demonstration included evaluating the sampler's ability to (1) consistently collect a given volume of sediment, (2) consistently collect sediment in a given depth interval, (3) collect samples with consistent characteristics from a homogenous layer of sediment, (4) collect a representative sample from a clean sediment layer below a contaminated sediment layer, and (5) be adequately decontaminated. Additional primary objectives were to measure sampling time and estimate sampling costs. Secondary objectives included (1) documenting the skills and training required for sampler operation, (2) evaluating the sampler's ability to collect samples under a variety of site conditions, (3) assessing the sampler's ability to collect an undisturbed

sample, (4) evaluating sampler durability, and (5) documenting the availability of the sampler and its spare parts. To ensure data usability, data quality indicators for precision, accuracy, representativeness, completeness, and comparability were also assessed based on project-specific QA objectives.

The Russian Peat Borer was demonstrated at sites in EPA Regions 1 and 5. At the Region 1 site, the sampler was demonstrated in a lake and wetland. At the Region 5 site, the sampler was demonstrated in a river mouth and freshwater bay. Collectively, the two sites provided multiple sampling areas with the different water depths, sediment types, sediment contaminant characteristics, and sediment thicknesses necessary to properly evaluate the sampler. Based on the predemonstration investigation results, demonstration objectives, and site support facilities available, (1) the Hand Corer was used as the reference sampler in the lake, wetland, and freshwater bay and (2) the Vibrocorer was used as the reference sampler in the river mouth. A complete description of the demonstration and a summary of its results are available in the "Innovative Technology Verification Report: Sediment Sampling Technology—Aquatic Research Instruments Russian Peat Borer" (EPA/600/R-01/010).

TECHNOLOGY DESCRIPTION

The Russian Peat Borer is a manually driven, chambered-type, side-filling core sampler designed to collect discrete, relatively uncompressed sediment samples. Sampler components include a stainless-steel core tube, aluminum extension rods, a stainless-steel turning handle, and a Delrin® core head and bottom point that support a stainless-steel cover plate. The cover plate and bottom point are sharpened to minimize sediment disturbance during sampler deployment. The core tube is hinged to the cover plate by two pivot pins at the top and bottom of the plate. Support equipment for the sampler may include a slide-hammer mechanism to aid sampler deployment and retrieval in consolidated sediment. To collect a sediment sample, the Russian Peat Borer is manually inserted into sediment, and the core tube is turned 180 degrees clockwise. This procedure allows the core tube to rotate and its sharp edge to longitudinally cut through the sediment, collecting a semicylindrical sediment core. While the core tube is manually turned, the stainless-steel cover plate provides support so that the collected material is retained in the core tube.

VERIFICATION OF PERFORMANCE

Key demonstration findings are summarized below for the primary objectives.
Consistently Collecting a Given Volume of Sediment: In the shallow depth interval (0 to 4 inches below sediment surface [bss]), to collect a specified number of samples, the Russian Peat Borer required 33 percent more attempts than expected (65 actual versus 49 expected), whereas the reference samplers required 14 percent more attempts than expected (49 actual versus 43 expected). In the moderate depth interval (4 to 32 inches bss), the Russian Peat Borer required 21 percent more attempts than expected (46 actual versus 38 expected), but the reference samplers required 156 percent more attempts than expected (64 actual versus 25 expected).

For the shallow depth interval, mean sample recoveries ranging from 71 to 84 percent were achieved by the Russian Peat Borer, whereas mean sample recoveries for the reference samplers ranged from 85 to 100 percent. The variation in sample recoveries as measured by their relative standard deviations (RSD) ranged from 26 to 42 percent for the Russian Peat Borer, whereas the reference samplers' RSDs ranged from 0 to 33 percent. For the moderate depth interval, mean sample recoveries ranging from 75 to 101 percent were achieved by the Russian Peat Borer, whereas the reference samplers' mean sample recoveries ranged from 21 to 82 percent. The RSDs for the Russian Peat Borer ranged from 6 to 31 percent, whereas the reference samplers' RSDs ranged from 3 to 161 percent. (Note: sample recoveries exceeding 100 percent resulted from the volumetric measurement error associated with the presence of void spaces when the sediment was transferred to a graduated container.)

Consistently Collecting Sediment in a Given Depth Interval: The Russian Peat Borer collected samples in all depth intervals and demonstration areas, which contained various sediment types. The reference samplers were unable to collect samples in the deep depth interval (4 to 11 feet bss). For the shallow depth interval, the Russian Peat Borer's actual core lengths equaled the target core length in 98 percent of the total sampling attempts. The reference samplers' actual core lengths equaled the target core length in 94 percent of the total sampling attempts. However, the results for the samplers were significantly different for the moderate depth interval: 93 percent for the Russian Peat Borer compared to 13 percent for the reference samplers.

Collecting Samples with Consistent Characteristics from a Homogenous Layer of Sediment: Based on particle size distribution results, both the Russian Peat Borer and reference samplers collected samples with consistent physical characteristics from two homogenous layers of sediment (a sandy silt layer and a clayey silt layer).

Collecting a Representative Sample from a Clean Sediment Layer Below a Contaminated Sediment Layer: The Russian Peat Borer collected samples from a clean sediment layer below a contaminated sediment layer that were at least as representative as the samples collected from the clean layer by the reference sampler (the Hand Corer); contaminant concentrations in the samples collected by both samplers were not statistically different at a significance level of 0.05.

Sampler Decontamination: Both the Russian Peat Borer and reference samplers demonstrated the ability to be adequately decontaminated after sampling in areas contaminated with either polychlorinated biphenyls or arsenic.

Sampling Time: Compared to the reference samplers, the Russian Peat Borer not only was able to collect samples in all depth intervals and demonstration areas but also reduced sampling time by 16 to 77 percent, depending on the area.

Sampling Costs: Of the sampling costs estimated for two of the four areas sampled, in one area the sampling costs for the Russian Peat Borer were 90 percent less than those for the reference sampler (the Vibrocorer), and in the other area the sampling costs for the Russian Peat Borer were 22 percent more than those for the reference sampler (the Hand Corer). Key demonstration findings are summarized below for the secondary objectives.

Skill and Training Requirements: The Russian Peat Borer, like the Hand Corer, is easy to operate and requires minimal skills and training. However, operation of the Vibrocorer is relatively complicated and requires moderate skills and training. The Russian Peat Borer was operated by one person, whereas the Hand Corer was operated by one or two persons and the Vibrocorer was operated by two persons. When more than two extension rods were required, the Hand Corer was operated using a tripod-mounted winch. The Vibrocorer operation required a motor-operated winch, whereas the Russian Peat Borer was operated without a winch throughout the demonstration.

Sampling Under a Variety of Site Conditions: The Russian Peat Borer collected samples in all depth intervals and demonstration areas, which contained various sediment types. The reference samplers were unable to collect samples in the deep depth interval (4 to 11 feet bss). Neither the Russian Peat Borer nor the Hand Corer requires a power supply. In contrast, the Vibrocorer requires a three-phase, 230- or 440-volt, 50- to 60-hertz power supply, which is a sampler limitation if the power supply fails.

Collecting an Undisturbed Sample: The Russian Peat Borer collected representative core samples of consolidated sediment in discrete depth intervals. Visual observations indicated that these samples were relatively uncompressed. In addition, the Russian Peat Borer collected sediment samples containing live biota. The reference samplers collected relatively compressed core samples of both consolidated and unconsolidated sediments from the sediment surface downward. In moderate and deep depth intervals, samples collected by the reference samplers may be of questionable representativeness because of core shortening and core compression. In the samples collected by the Russian Peat Borer, sediment stratification was preserved for consolidated sediment but not for unconsolidated sediment. Sediment stratification was preserved for both consolidated and unconsolidated sediments in the samples collected by the reference samplers.

Sampler Durability and Availability: Based on their materials of construction and engineering designs, both the Russian Peat Borer and reference samplers are considered to be sturdy. The Russian Peat Borer and its support equipment are not expected to be available in local retail stores. Similarly, the primary components of the Hand Corer and Vibrocorer are not expected to be available in local retail stores; extension rods for the Hand Corer may be locally available.

Based on the demonstration results, the Russian Peat Borer can be operated by one person with minimal skills and training and does not require support equipment such as a winch and power source even when collecting sediment samples at depths up to 11 feet bss. The sampler can collect representative and relatively uncompressed samples of consolidated sediment in discrete depth intervals. The sampler preserves sediment stratification in consolidated sediment samples, but sediment stratification may not be preserved in unconsolidated sediment samples. The Russian Peat Borer is a superior alternative to conventional sediment samplers, particularly for sampling consolidated sediment. As with any sampler selection, the user must determine the appropriate sampler for a given application based on project-specific data quality objectives.

Gary J. Foley, Ph.D.
Director
National Exposure Research Laboratory
Office of Research and Development

NOTICE: EPA verifications are based on an evaluation of technology performance under specific, predetermined criteria and appropriate quality assurance procedures. The EPA makes no expressed or implied warranties as to the performance of the technology and does not certify that a technology will always operate as verified. The end user is solely responsible for complying with any and all applicable federal, state, and local requirements.

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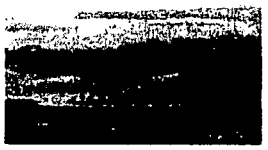

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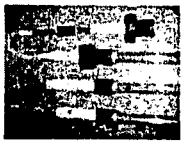
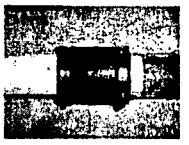
Universal Percussion Corer

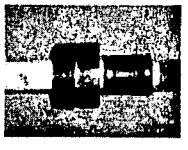
UNIVERSAL CORE HEAD



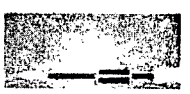
- Polyethylene, polycarbonate and stainless steel construction
- one-way check valve core retainer
- rubber coupler sleeve
- 10m x 10mm diam
- polyester lowering line
- linereel
- one-ea. thin-walled-clear polycarbonate core barrel (68 mm x 71 mm x 120 cm)
- "T"-handle, core extruding plug
- poly end caps
- spares
- toolbox carry case
- hexdriver, complete: $295.00



- Bronze Gravity Weights, 4 kg each (five-max. capacity) complete: $59
- Aluminum holobar extension rod w/ stainless steel couplers (40mm diam. x 2.4m): $122
- Aluminum holobar extension rod w/ stainless steel couplers (40mm diam. x 1.2m): $69
- Slide hammer assembly, bronze and stainless steel construction: $105
- Clear polycarbonate core barrel with poly end caps: 68 mm x 71 mm x 240 cm: $75
- Clear polycarbonate core barrel with poly end caps: 68 mm x 71 mm x 120 cm: $45
- Clear polycarbonate core barrel with poly end caps: 68 mm x 71 mm x 60 cm: $25
- Polyester Lowering Line x 10mm diam. X 10 m: $25



Notes: Simple, versatile, and easy to use. Thin-wall core barrels and positive check



valveassembly collects quality samples in a variety of sediments and conditions. An excellent quality brass check valve provides good flushing and positive vacuum to retain cores during recovery, *without core catchers*.

Upon retrieval, one must plug the bottom of the core tube before breaking the air-water interface (with core extruding plug or poly end cap) to prevent loss of sample. Clear polycarbonate core barrel material readily available at most plastic distributors.

Core extruding plugs allow one to incrementally extrude sediments upward with aid of a Core Extruding Apparatus. Core barrels can easily be drilled/plugged (for pore water extraction), serrated, cut and split to meet the special needs of the investigation.

The Universal Core Head can be deployed in a variety of ways:

1. **In shallow lakes:** Aluminum holobar extension rods to maximum water depth up to 15m. Provides: good "feel" for the sediments, good control of penetration depth, long cores of the water-sediment interface.
2. **In deep lakes:** Polyester Lowering Line with Bronze Gravity Weights and/or Slide hammer assembly. Sampler is gently lowered into the sediments to recover undisturbed samples. For deeper penetration, the slide hammer assembly can be raised and dropped (on a separate, small diam. wire, not included) "T"-handle.
3. **In shallow water or SCUBA:** "T"-handle with or without a Slide hammer assembly can be used in shallow marsh and wetland conditions.

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APPENDIX B

SAMPLING AND ANALYSIS PLAN / VOLUME II - QUALITY ASSURANCE PROJECT PLAN

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SAMPLING AND ANALYSIS PLAN
VOLUME II - QUALITY ASSURANCE PROJECT PLAN
KOPPERS POND
KENTUCKY AVENUE WELLFIELD SITE
OPERABLE UNIT 4
HORSEHEADS, NEW YORK

_____

Prepared by: (Preparer's Name and Organization Affiliation)

_____

Address and Telephone Number

_____

Day/Month/Year

Project Manager: _____

Signature

_____

Printed Name/Date

QA Officer: _____

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USEPA QA Officer Approval: _____

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SAMPLING AND ANALYSIS PLAN
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KOPPERS POND
KENTUCKY AVENUE WELLFIELD SUPERFUND SITE
OPERABLE UNIT 4
HORSEHEADS, NEW YORK

1.0 INTRODUCTION

On behalf of the Koppers Pond RI/FS Group (the Group), Cummings/Riter Consultants, Inc. (Cummings/Riter), with the assistance of AMEC Earth and Environmental, Inc. (AMEC), has prepared this Sampling and Analysis Plan (SAP) to support remedial investigation (RI) activities to be conducted for Koppers Pond in the Village and Town of Horseheads, Chemung County, New York (the Site). Figure 1 of the Remedial Investigation/Feasibility Study (RI/FS) Work Plan shows the Site location. Pursuant to the Administrative Settlement Agreement and Order on Consent (Index No. CERCLA-02-2006-2025), Koppers Pond is being addressed as Operable Unit 4 of the Kentucky Avenue Wellfield Superfund Site.

The SAP consists of two plans: Volume I - Field Sampling Plan (FSP) and Volume II - Quality Assurance Project Plan (QAPP). This Appendix B is the QAPP, which is to be used in conjunction with the FSP (Appendix A) to support activities related to the performance of the RI/FS. This QAPP has been prepared in accordance with the following U.S. Environmental Protection Agency (USEPA) guidance documents:

- *Data Quality Objectives Process for Hazardous Waste Site Investigations* (EPA QA/G-4HW), January 2000;
- *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations* (EPA QA/R-5), March 2001;
- *Guidance for Quality Assurance Project Plans* (EPA QA/G-5, December 2002; and

- *Uniform Federal Policy for Quality Assurance Project Plans, Parts 1, 2, and 3* (EPA-505-B-04-900A, B, and C), March 2005.

The QAPP describes the RI activities involved with the acquisition of environmental data, whether generated from direct measurements, collected from other sources, or compiled from computerized databases and information systems. The purpose of this QAPP is to document the results of the technical planning process; provide a clear, concise, and complete plan for the environmental data operation and its quality objectives; and identify key project personnel. The QAPP integrates quality assurance and quality control (QA/QC) with the environmental data operation to assure that the results obtained are of the type and quality needed and expected.

This QAPP identifies samples designated for analysis, analytical parameters, analytical methods, and procedures used to ensure that the data quality objectives (DQOs) are achieved, including procedures for sample transportation, analysis, validation, and reporting. These procedures are to be followed during field activities and laboratory analyses conducted in support of the RI for the Site.

Figure B1-1 provides a project schedule for the performance of the activities described in this QAPP.

1.1 CORPORATE QUALITY MANAGEMENT

The Cummings/Riter Corporate QA Program establishes policies and procedures for personnel engaged in project work, and site-specific QAPPs are prepared to ensure that the data collected are adequate for the intended purpose. The Corporate QA Program ensures that project tasks and deliverables are completed in a manner that meets quality standards established based on the American National Standard Institute (ANSI) and American Society for Quality Control (ASQC) *Quality Systems--Model for Quality Assurance in Design/Development, Production, Installation, and Servicing* (ANSI/ASQC Q91-1987) and *Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs* (ANSI/ASQC E4-1994), as they apply to the scope of services provided. This standard is technically equivalent to

the International Standard Organization (ISO) 9001 and meets the requirements of *EPA Requirements for Quality Management Plans* (EPA QA/R2). The Corporate QA Program provides a means of control and review for the following:

- Project organization, responsibilities, and personnel qualifications and training requirements;
- Development and documentation of work procedures;
- Field investigation and testing, including performance, equipment calibration, sample control, documentation, and verification;
- Analysis and design, including performance, documentation, verification, and reporting;
- Procurement planning, documentation, and review;
- Records administration, including control and retention;
- QA activities, including auditing;
- Nonconformance, including identification, documentation, and reporting; and
- Quality improvement.

The Corporate QA Program is documented in the Cummings/Riter *Quality Assurance Manual* (Cummings/Riter, 1993), which describes in detail the use of such tools as audit procedures and standard operating procedures (SOPs), as well as the implementation process and lines of responsibility.

The AMEC Quality Management Plan is based on ANSI/ASQC E4-2004 and EPA QA/R-2. As described in EPA QA/R-2, the following key elements have been incorporated into the AMEC Quality Management Plan:

- Mission and quality policy of the organization;
- Specific roles, authorities, and responsibilities of management and staff with respect to QA/QC activities;
- Means by which effective communications with personnel actually performing the work are assured;
- Processes used to plan, implement, and assess the work performed;
- Process by which measures of effectiveness for QA/QC activities will be established and how frequently effectiveness will be measured; and
- Continual improvement based on lessons learned from previous experience.

1.2 BACKGROUND

Koppers Pond consists of an approximately eight-acre, "V" shaped warm-water pond with typical water depths of approximately three to six feet. At normal stage, the surface water elevation is 887± feet above mean sea level. The pond receives inflow at the northern end of its western leg from the Industrial Drainageway, a surface water course that originates at the outlet of a 74-inch diameter underground pipe (Chemung Street Outfall), approximately 2,300 feet to the northwest. The Industrial Drainageway receives permitted process discharges originating at the former Westinghouse Horseheads plant site and surface runoff from a contributory watershed area of approximately 604 acres. Discharge from Koppers Pond flows into two outlet streams at its southern end, which then converge approximately 500 feet downstream to form the outlet channel. The flow eventually converges with Halderman Hollow Creek, which in turn feeds into Newtown Creek, a primary tributary to the Chemung River. The Site background and description are summarized in the RI/FS Work Plan.

1.3 ORGANIZATION OF QAPP

Following this introduction, Section 2.0 describes the anticipated RI sampling and analytical activities; Section 3.0 describes the project organization and responsibilities; Section 4.0 presents the QA objectives for measurement data; Section 5.0 describes custody procedures for collected samples; Section 6.0 references sampling procedures; Section 7.0 provides calibration procedures and frequency requirements; Section 8.0 identifies analytical procedures; Section 9.0 presents required internal QC checks; Section 10.0 describes data reduction, validation, and reporting requirements; Section 11.0 presents performance and system audit procedures; Section 12.0 describes preventative maintenance requirements; Section 13.0 describes measurement performance criteria and procedures to be used to ensure that data acquired during the RI is useable; and Section 14.0 describes corrective action procedures.

2.0 PROJECT DESCRIPTION

This section identifies the media to be sampled and the analytical parameters for those samples. Sample analytical results will be used to achieve the data quality objectives (DQOs) and RI objectives identified in Section 5.0 of the RI/FS Work Plan.

2.1 CONSTITUENTS OF POTENTIAL CONCERN IN ENVIRONMENTAL MEDIA

In 1994 and 1995, the RI for Operable Unit 3 of the Kentucky Avenue Wellfield Site included two rounds of surface water and sediment sampling of Koppers Pond to help identify constituents of potential concern (COPCs). In 1998, CDM Federal Programs Corporation (CDM) collected water quality data and sediment samples in support of a baseline ecological risk assessment of Koppers Pond (CDM, February 1999).

The historical data were collected when treated industrial waste waters were being discharged from the former Westinghouse Horseheads plant site at rates significantly greater than current process water discharges. Because such discharges have since been substantially reduced, the reported data from these prior studies may not be representative of current conditions. In addition, historical investigations produced very limited vertical profiling (i.e., stratified) data for COPCs in sediment. The RI will include additional surface water and sediment sampling to provide data that are more recent and vertical profiling of COPC concentrations in sediment at select locations. Table B2-1 summarizes COPCs for surface water and sediment based on historical results. The rationale for selection of the COPCs is provided below.

2.1.1 Surface Water

Metals detected in historical surface water samples reflect the influence of the permitted treated waste water discharges to the Industrial Drainageway, including aluminum, chromium, lead, zinc, and fluoride. Pesticide compounds α-BHC (benzene hexachloride) and β-BHC were detected in some surface water samples collected in 1994.

The available data from historical studies indicate that COPCs are not present in Koppers Pond surface water at concentrations that could cause or contribute to unacceptable human health risk. The 1995 baseline human health risk assessment (CDM, November

1995), using data from 1994 and 1995, showed human health risks associated with surface water pathways to be less than 10^{-6}, the lower end of target risk range as defined in USEPA regulations and guidance.

There could be some potential for re-dissolution of COPCs from sediments into surface water as the chemistry of the pond adjusts to the reduction of process waste water discharges. In addition, the effects (if any) of the observed floc associated with past discharges has not been fully evaluated. Surface water data are not available to draw conclusions with respect to potential ecological risk. In prior sampling, certain metals were found at concentrations above applicable New York State Class C ambient water quality criteria, but the current concentrations are not known. Accordingly, metals are considered a COPC for surface water in Koppers Pond. Also, even though the source is likely to be an historic area-wide application, the pesticide compounds α-BHC and β-BHC cannot be ruled out at this time as COPCs for Koppers Pond surface water and as potential contributors to ecological risks.

2.1.2 Pond Sediments

Pond sediments are the primary affected environmental medium at Koppers Pond. Metals and hydrophobic organic compounds such as polychlorinated biphenyls (PCBs), pesticides, and polycyclic aromatic hydrocarbons (PAHs) may adsorb onto sediments and potentially become available to the aquatic food web. Historical sediment data show the presence of several metals at concentrations above New York State Department of Environmental Conservation (NYSDEC) (January 1999) sediment screening levels, including antimony, arsenic, cadmium, chromium, copper, lead, manganese, mercury, nickel, silver, and zinc. While historical data have not shown significant detections of volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs) detected in pond sediment samples consisted primarily of PAHs and, to a lesser degree, phthalates. Laboratory estimated concentrations of at least one PAH exceeded NYSDEC sediment screening levels in one pond sample (SD-17) and one outlet channel sample (SD-20B). Pesticides were also found in various sediment samples, several of which were detected in at least one sample above NYSDEC sediment screening levels. In addition, PCB Aroclor 1254 was detected in approximately two-thirds of historical sediment samples collected from Koppers Pond and its outlet channels at concentrations exceeding NYSDEC sediment screening levels.

Toxicity testing of Koppers Pond sediments using the *Hyalella azteca* (amphipod) 10-day survival test and the *Chironomus tentans* (midge) 10-day survival and growth test was also conducted. This testing did not show the level of toxicity that might have been expected based on the chemical profile of these sediments.

In the 1995 baseline human health risk assessment, direct exposure to Koppers Pond sediments did not pose an unacceptable potential risk. The potential human health risk associated with consumption of fish taken from Koppers Pond was, however, estimated to be above USEPA's acceptable risk range. The majority of the potential incremental excess lifetime cancer risk is attributable to PCBs, although arsenic also contributes to the total potential cancer risk.

The draft baseline ecological risk assessment (CDM, February 1999) showed certain pesticides, metals, and PCBs in sediment potentially contributing to unacceptable ecological risk. The chemical data on the sediments are not corroborated by acute toxicity testing that showed either no or very limited acute toxicity of sediments to aquatic organisms, nor are the risk assessment models consistent with actual measurements of COPC levels in aquatic organisms. Resolution of these issues will require further evaluation in the RI. At present, pesticides, PCBs, and heavy metals are considered to be the COPCs for sediments in Koppers Pond. Although some PAHs have been detected in sediments, these appear to be of lesser consequence, on the basis of lower concentrations, less pervasive occurrence, and the absence of PAHs in fish tissue samples analyzed as part of the Operable Unit 3 RI (Philip Environmental Services Corporation, March 1996).

2.2 SCOPE OF WORK

RI activities include the collection of environmental data to support the evaluation of appropriate remedial alternatives for the Site. The field activities related to data collection during the RI phase are as follows:

- Task 3.1 - Surveying and Mapping
  - Establish survey control
  - Install pond staff gauge

- Task 3.2 - Surface Water and Sediment Sampling
  - Collection of pond and outlet channel surface water samples
  - Collection of pond and outlet channel sediment samples

- Task 3.3 - Pond Bathymetry
  - Conduct pond bathymetry survey (navigable portion) using Global Positioning System (GPS) and echo sounder
  - Collect pond depth measurements manually in shallow and other non-navigable areas

- Task 3.4 - Assess Sediment Thickness
  - Measure sediment thickness at each sediment sampling location
  - Collect additional measurements if sediment thickness is non-uniform

- Task 3.5 - Assess Potential Ongoing Sources
  - Collect surface water samples and flow readings of the barrier well treated water discharge and the Cutler-Hammer Division of Eaton Corporation (Cutler-Hammer) discharge at the former Westinghouse Horseheads plant site
  - Conduct video survey of underground piping upstream of the Chemung Street Outfall
  - Collect sample of floc if present in underground piping
  - Collect a surface water sample of the Industrial Drainageway at the Chemung Street Outfall
  - Perform field reconnaissance of potential storm water inflows to the Industrial Drainageway and Koppers Pond
  - Collect samples of any identified significant points of storm water inflow
  - Inspect north shore of pond and northeast bank of lower drainageway adjacent to the Old Horseheads Landfill for visual indications of seeps
  - Sample any identified seeps associated with the Old Horseheads Landfill and draining to Koppers Pond

- Task 3.6 - Assess Pond Hydrology
  - Collect measurements of pond surface elevation
  - Collect measurements of nearby groundwater elevations

- Task 3.7 - Fish Tissue Sampling
  - Collect fish specimens for laboratory analysis

In addition, local water bodies exhibiting physical characteristics similar to those of Koppers Pond, but not potentially affected by Site sources, will be identified as candidate reference ponds. The need for investigation of such reference ponds (e.g., sediment sampling, fish tissue sampling) will be determined following the review of RI data and the comparison of these data to prior sampling results for Koppers Pond.

Table B2-2 presents a summary of the RI sampling program, and Table B2-3 presents the sample preparation and holding time requirements for these samples. Following are descriptions of sample frequencies and analytical requirements.

2.2.1 Surface Water, Sediment, and Potential Source Sampling

Surface water and sediment samples will be collected at a total of 17 locations within Koppers Pond, the West Outlet, the East Outlet, and the Outlet Channel. The proposed sampling locations are shown on Figure A-1 of the FSP.

The investigation of potential ongoing sources to Koppers Pond will involve the sampling of the barrier well treatment facility and the Cutler-Hammer process water discharge from the former Westinghouse Horseheads plant site. In addition, potential ongoing sources identified from field reconnaissance will be sampled. Such potential sources include significant surface water discharges contributing to the Industrial Drainageway and seeps associate with the Old Horseheads Landfill.

Surface water, sediment, and aqueous potential source samples will be analyzed for the CLP target compound list (TCL) VOCs, SVOCs, and pesticides/PCBs and for target analyte list (TAL) inorganic parameters. Aqueous samples collected for TAL analysis will be analyzed for both the total and dissolved fractions of metals to allow for direct comparisons to ambient water quality criteria. Surface water samples will also be analyzed for the general chemistry parameters ammonia, fluoride, hardness, nitrites, and total suspended solids. Dissolved oxygen, pH, oxidation-reduction potential (ORP), temperature, and specific conductance will be determined in the field at the time of sampling. Where applicable, flow rates will be recorded from available flow meters (e.g., barrier well discharge) or estimated from field observations.

Sediment samples will also be analyzed for total organic carbon, and select sediment samples will be tested for grain-size determination and acid volatile sulfide and simultaneously extracted metals (AVS/SEM). pH and ORP will be recorded for sediment samples at the time of field sampling.

Sediment and water sample analyses will be conducted using SW-846 methods for Contract Laboratory Program (CLP) TCL and TAL parameters, as identified in Table B2-4 for organics and Table B2-5 for remaining parameters. Non-CLP parameters (e.g., general chemistry for aqueous samples and lipids for fish tissue) will also be analyzed according to the methods identified in Tables B2-4 and B2-5.

During videotaping of the Chemung Street Outfall, the viability of floc sampling will be evaluated. If floc is visible adhering to the pipe walls, samples will be collected and analyzed for TCL organics and TAL inorganics using SW-846 methods.

2.2.2 Fish Tissue Sampling

Fish sampling will be used to provide current data on PCB and metals concentrations in fish tissue for evaluation of risks to human and ecological receptors and allow for comparison of current conditions to those determined in the 2003 sampling event (Civil & Environmental Consultants, Inc., July 2003). Elements of the RI fish sampling program were developed based on the objectives of the refined risk assessment, discussions with agency personnel, and a review of relevant agency documents, including the USEPA November 2000 *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Third Edition* and the NYSDEC *Draft Procedure for Collection and Preparation of Aquatic Biota for Contaminant Analysis* (October 2002). Analytical methods are identified in Table B2-4 for organic parameters and in Table B2-5 for other parameters.

The laboratory will process and prepare fish samples (e.g., weighing, filleting, homogenizing) in accordance with accepted protocols (NYSDEC, October 2002). Following preparation, fish samples will be analyzed for TCL pesticides/PCBs, TAL metals, and lipid content. In addition, the collected fish will be examined in the field for any external signs of deformities, tumors, or lesions. If such deformities are noted, suggesting the potential for PAH impacts, equal numbers of individual fish showing

deformities, tumors, or lesions and individual fish without deformities, will be analyzed for TCL SVOCs. The lipid content of fish samples will be determined to facilitate the evaluation of the concentrations of lipid-soluble constituents (e.g., PCBs).

3.0 PROJECT ORGANIZATION AND RESPONSIBILITY

This section provides a description of the RI/FS project organization and the responsibilities associated with each of the positions in this organization. The project organization is represented on Figure B3-1. A distribution list of the QAPP recipients is provided as Table B3-1.

3.1 USEPA PROJECT MANAGER

Ms. Isabel Rodrigues is the Remedial Project Manager for USEPA. She will coordinate USEPA activities during the RI/FS and will be the point of contact for USEPA.

3.2 PROJECT COORDINATOR

Mr. Leo M. Brausch, P.E., will serve as the Project Coordinator for the Group. Mr. Brausch will be the primary contact between the Group and USEPA and will monitor the project technical performance, schedule, and budget.

3.3 PROJECT MANAGERS

Mr. William Smith, P.E., will serve as the Cummings/Riter Project Manager. Mr. Smith will be the primary contact between Cummings/Riter and the Project Coordinator. He will also be responsible for all Cummings/Riter technical, financial, and scheduling matters.

Mr. John Samuelian, Ph.D., will serve as the AMEC Project Manager. Mr. Samuelian will be the primary contact between AMEC and the Project Coordinator and will be responsible for review of data generated for risk modeling, coordination of risk evaluations, and preparation of the supplemental baseline ecological risk assessment and updated baseline human health risk assessment.

3.4 PROJECT SUPERVISOR

Mr. Bruce Geno will serve as the Cummings/Riter Project Supervisor. Mr. Geno will direct the field sampling crews, interface with the laboratory subcontractor, and be responsible for preparation of project documents.

3.5 QUALITY ASSURANCE OFFICER AND HEALTH AND SAFETY COORDINATOR

Ms. Denise Ladebauche will serve as the QA Officer. She is responsible for reviewing the SOPs; reviewing field and laboratory data for compliance with QA objectives (i.e., precision, accuracy, comparability, and completeness); and reporting deficiencies to project management.

Mr. Kenneth Bird, C.I.H., of Cummings/Riter will serve as the Health and Safety Coordinator. He is responsible for reviewing the SOPs and modifications to the Health and Safety Plan (HASP), as needed, based on field monitoring results.

3.6 SITE SAFETY OFFICER

[To be determined.] The Site Safety Officer will be responsible for implementation of the HASP and documenting health and/or safety issues that arise during completion of the RI tasks.

3.7 PROJECT/FIELD TEAM MEMBERS

The field investigation will be completed as a collaborative effort using personnel from both Cummings/Riter and AMEC. Cummings/Riter will provide the on-Site field team supervisor and coordinate sampling and surveying activities. AMEC personnel will perform field investigation activities associated with fish-tissue sampling.

3.8 ANALYTICAL LABORATORY

TestAmerica Laboratories, Inc. (TestAmerica) (formerly known as Severn Trent Laboratories, Inc.) will provide analytical laboratory services for the Koppers Pond RI under contract to the Group. An analytical laboratory project manager will be identified from TestAmerica's Pittsburgh, Pennsylvania laboratory and will be responsible for execution of the analytical testing program for the project. The name of the laboratory project manager will be provided upon request prior to sample collection. The laboratory project manager will be responsible for ensuring that laboratory internal QA procedures are followed and will be the point of contact for the Project Coordinator, Project Supervisor, and QA Officer.

3.9 OTHER CONTRACTORS

Other contractors to be utilized for RI project activities include the following:

- Fagan Engineers, P.C., Elmira, New York – certain surveying tasks and technical and logistical support for field activities;
- Piping contractor – video survey of underground piping leading to the Chemung Street Outfall; and
- Geotechnical laboratory – grain-size determinations of select sediment samples.

In addition to the human health and ecological risk evaluations, AMEC will conduct the independent validation of the analytical laboratory data.

4.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

4.1 QA OBJECTIVES

The overall QA objective for the RI/FS is to develop procedures which, when followed properly, will provide assurance that reasonable decisions based on laboratory and field data generated during the investigations are technically sound, statistically valid, and properly documented. Data will ultimately be used to allow assessment of potential human health and ecological risks associated with Site-related COPCs in Koppers Pond. Data generated during the RI will be compared to Site-specific or other risk-based cleanup levels.

The primary purpose of this section of the QAPP is to define statistical acceptance criteria for chemical data generated by the field sampling team and analytical laboratory. These statistically based criteria are referred to in this document as DQOs. In developing the DQOs, a series of planning steps was conducted based on the USEPA *Data Quality Objectives Process for Hazardous Waste Site Investigations* (EPA QA/G-4HW, January 2000) and the Settlement Agreement to ensure that the type, quantity, and quality of environmental data are appropriate for their intended use. These planning steps for DQO development are provided below.

4.1.1 DQO Planning Team

The DQO planning team consists of the USEPA, the Group, the Project Coordinator, Cummings/Riter, AMEC, and interested stakeholders. The Project Coordinator will offer technical advice to the planning team for DQO development. Because several members of the Group are municipal entities with publicly elected representatives in local government and owners of land surrounding the pond, stakeholder interests are well represented.

4.1.2 Problem Statement

The data obtained through implementation of the FSP will be used to provide current data regarding COPC concentrations in environmental media and fill data gaps remaining from prior studies as described in the Preliminary Conceptual Site Model (PCSM)

submitted to USEPA in February 2007. Existing surface water, sediment, and fish-tissue data were obtained at times when industrial discharges to the pond were significantly greater. Because Koppers Pond represents a dynamic environmental system, COPC concentrations may have changed with time, and sediments need to be sufficiently characterized to determine the current lateral and vertical extent of COPCs. The RI will address these data needs and will include efforts to identify ongoing sources of COPCs to Koppers Pond. Surface water, sediment, and fish-tissue data will be used for improved understanding of the current relationships among potentially impacted environmental media, migration pathways, and potential human and ecological receptors. Data from potential ongoing sources will be used to identify contributors to environmental impacts beyond what is currently known. Ultimately, RI data will be used to revise and supplement the findings of the baseline human health and ecological risk assessments for determination of the need for remedial action, and, if required, appropriate action levels and corresponding response actions.

4.1.3 Identification of the Decision

The RI data will be used to determine whether current COPC concentrations in Koppers Pond pose unacceptable risks to human health or the environment. If COPCs are determined to pose unacceptable risks, the results of the risk assessment and applicable or relevant and appropriate requirements will be used to establish cleanup goals. The RI data would then be used to identify the locations of environmental media containing concentrations of COPCs that exceed the cleanup goals to determine whether additional investigation or other response actions are necessary.

4.1.4 Identification of Inputs

A combination of sampling and modeling (i.e., risk assessment) will be used to resolve the decision statement. The PCSM summarizes analytical results and the practical concentration ranges for COPCs for previously collected data, and describes limitations of the data used for the human health and ecological risk assessments. Analytical methods and samples designated for analysis have been specifically selected with consideration of performance characteristics (precision, bias, and method detection limits, etc.), exposure pathways, and potential receptors to provide the measurements necessary to update the baseline risk assessment, compare concentrations of COPCs in environmental media with potential action levels, and help identify potential ongoing

sources of COPCs. To help assure useable data, the analytical laboratory (TestAmerica) is accredited through the National Environmental Laboratory Accreditation Program, which uses standards set by the National Environmental Laboratory Accreditation Conference, to perform the analyses on which resolution of the decision statement will be based. Analytical methods to be used and associated quantitation limits (QL) for the analytical parameters are provided in Tables B2-4 and B2-5 for organic and other parameters, respectively.

4.1.5 Study Boundaries

The spatial boundaries for the RI coincide with the watershed area contributing to the location of the most downstream surface water and sediment samples. Vertical profiling of sediment concentrations of COPCs will help establish the influence of historical activities within the watershed, while surface water sampling will help to assess ongoing sources of COPCs. It is noted that there may be practical constraints to sample collection in areas that are inaccessible by boat or are too soft to cross in waders. Water level measurements of Koppers Pond and in proximate groundwater monitoring wells will be used along with historical groundwater monitoring results to evaluate the pond-groundwater interaction and potential impact on surface water quality and COPC concentrations in sediment and surface water. Water level measurements will be recorded for a period of three months to correlate to historical groundwater monitoring data. The time period for which the environmental conditions observed and recorded are assumed to be representative of will be defined in the baseline risk assessment.

4.1.6 Decision Rule

For this step of DQO planning, the objectives are as follows:

- Specify the statistical parameters that characterize the population of interest;
- Specify the action level for the decision;
- Confirm that the action level is above measurement detection limits so that reliable comparisons can be made; and
- Combine the statistical parameter, the scale of decision-making, and the action level into an unambiguous decision rule that addresses the contamination problem.

Reported COPC concentration data may be statistically adjusted for input to the risk assessment model. For example, maximum concentrations, geometric mean concentrations, median concentrations, or percentile concentrations may be used as the statistical parameters of interest. Adjustments to reported COPC concentrations for input to the risk assessment model will be described in the RI/FS report. The risk assessment will establish numerical criteria for deciding whether Site COPC concentrations pose potential unacceptable risks. The RI/FS report will identify and evaluate appropriate remedial responses commensurate with assessed Site risks.

4.1.7 Decision Errors

The purpose of this step is to specify quantitative performance criteria for the decision rule expressed as probability limits on potential errors in decision-making. A decision error occurs when the data are misleading and result in choosing the wrong response action, in the sense that a different response action would have been chosen if access to "perfect data" or absolute truth were possible. The possibility of a decision error exists because the parameters of interest are estimated using data that are never perfect, but are subject to different variabilities at different stages of development, from field collection (i.e., sampling design error) to sample analysis (i.e., measurement error).

The possibility of making a decision error, although small, is undesirable due to the adverse consequences arising from that incorrect decision. A formal statistical decision procedure called hypothesis testing will be used to control the possibility of a decision error. Accordingly, data obtained from the RI will be used to choose between a presumed baseline environmental condition (i.e., COPCs in surface water and sediments pose unacceptable risks to potential receptors) and an alternative hypothesis (i.e., risks posed by COPCs in Site media are acceptable). The burden of proof will be placed on rejecting the baseline condition.

Probability limits on decision errors specify the level of confidence the site manager desires in conclusions drawn from Site data. False rejection and false acceptance decision error limits for the RI/FS have been set at the most stringent limits typically encountered for environmental data of 1 percent based on the consequences of making an incorrect decision. Performance in meeting these probability limits will be measured in terms of precision, accuracy, completeness, representativeness, and comparability.

4.1.7.1 Precision

Precision is defined as the degree of agreement between repeated measurements of the same parameter (i.e., reproducibility) under prescribed, similar conditions. Field and laboratory precision will be determined through the use of field duplicates, matrix spike/matrix spike duplicates (MS/MSD), and duplicate QC samples. For laboratory analytical parameters, the criteria for precision are defined by the analytical method.

Field Precision: The precision of field measurements will be based on standard deviation of a set of replicate measurements. For at least 5 percent of all samples collected during water sampling activities, measurements of dissolved oxygen, pH, ORP, temperature, and specific conductance will be performed at least three times on different aliquots of water. The same procedure will be applied to the field measurements of pH and ORP for sediments. Precision criteria for field measurements are shown in Table B4-1.

Laboratory Precision: One duplicate sample will be collected for every 20 surface water and sediment samples submitted for laboratory analysis or, if less than 20 samples are collected, then 1 duplicate per medium. An advisory limit of ±20 percent relative percent difference will be used for duplicate sample results that are greater than five times the QL (Tables B2-4 and B2-5). An advisory limit of ±QL will be used for duplicate sample results that are less than five times the QL.

MS/MSD samples will be collected once for every 20 samples for each medium (i.e., surface water, sediment, or fish) or, if less than 20 samples are collected, then 1 per medium. Percent recovery values for these samples will be compared to acceptance criteria provided in the analytical method.

4.1.7.2 Accuracy

Accuracy is the measure of the degree of agreement between an analyzed value and the true or accepted value where it is known (e.g., spike recovery). Field and laboratory accuracy will be monitored using field equipment blanks, trip blanks, and standards of known concentrations or values spiked into select samples.

Field Accuracy: Field measurements will be made for dissolved oxygen, pH, ORP, temperature, and specific conductance. Water levels in monitoring wells will also be measured. Water depth measurements during the bathymetric survey will be performed using an echo sounder. Field measurement, calibration, and maintenance procedures are described in the FSP. Accuracy criteria for field measurements are shown in Table B4-1. The accuracy of data produced by field instruments will be maintained and documented by performing initial calibrations followed by continuing calibration verifications and/or continuing calibrations with known standards.

One field equipment blank sample will be analyzed for each matrix type (surface water, sediment, biota) and for every batch of samples or every 20 samples analyzed, whichever is more frequent. Field equipment blank samples will be analyzed to check for procedural contamination and ambient conditions at the Site that may result in sample contamination.

The accuracy of pH measurements will be assessed by performing two pH measurements on a standard buffer solution (i.e., pH 4.0, pH 7.0 or pH 10.0, standard units). Each measurement must be within ±0.1 pH units of the standard, or the instrument will be recalibrated. Between measuring each replicate, the electrode will be withdrawn and rinsed with distilled water. This calibration verification will be performed after the collection of 20 samples or every four hours.

The echo sounder will be calibrated by using a portable depth gauge at two locations and adjusting the echo sounder to equate to the manual readings. The echo sounder will be integrated with differential global positioning system (GPS) survey equipment to assign horizontal positions to the depth data. Horizontal position accuracy will be approximately ±1 foot, and vertical accuracy will be approximately ±0.1 foot.

The accuracy of specific conductance measurements will be assessed by performing measurements on one calibration standard. Each measurement must be 10 percent of the standard, or the instrument will be recalibrated. Calibration will be checked after every 20 samples collected.

Temperature will be measured using a thermocouple on the pH meter. Acceptable accuracy is considered ±10 percent of the standard value. Temperature readings from the pH meter will be checked every other day with a National Institute of Standards and Technology (NIST) traceable thermometer.

The accuracy of dissolved oxygen measurements will be assessed by verifying calibration at the beginning of the day and every three hours of sample collection. A sample with zero dissolved oxygen will be prepared for the calibration check. Each measurement must be between 0.0 and 0.1 part per million, or recalibration will be required.

The accuracy of ORP measurements will be assessed by performing measurements on a calibration standard. The accuracy of ORP for instruments will be verified to within 10 percent the calibration standard, or recalibration will be required. Calibration verification will be performed for every 20 samples collected or every three hours.

Static water levels in monitoring wells will be measured using an electronic water sounder accurate to 0.01 foot. The sounder will be calibrated with a steel tape before it is shipped to the Site.

Laboratory Accuracy: Measures to be taken by the analytical laboratory to ensure accuracy include instrument tuning, instrument calibrations (initial and continuing), analyses of laboratory standards, and analyses of independent QC samples supplied by USEPA or traceable to the NIST.

4.1.7.3 Completeness

Completeness is defined as the measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. Data completeness will be expressed as the percentage of valid data obtained from the measurement system. For data to be considered valid, it must meet all the acceptance criteria including accuracy and precision, as well as any other criteria required by the prescribed analytical method.

It is anticipated that 100 percent of the proposed surface water and sediment samples can be collected. For the fish collections, a completeness of 80 percent is anticipated,

primarily due to uncertainty in sample collection success for the different species. It is expected that the laboratory will provide data meeting QC completeness criteria for at least 90 percent of the samples analyzed.

Field Completeness: Field measurements, including dissolved oxygen, pH, ORP, temperature, and specific conductance, will be taken where applicable. To ensure the completeness of the data generated from these measurements, calibration logs will be maintained for each instrument. In turn, these logs will provide an indication of whether any measurements were made with instruments that were not calibrated or functioning improperly.

Laboratory Completeness: Control limits for all chemical analyses performed by the analytical laboratory have been established. Analyses not meeting specified control limits for a particular analysis will be flagged. It is expected that the laboratory will provide data meeting QC completeness criteria for at least 90 percent of the samples analyzed.

4.1.7.4 Representativeness

Representativeness is understood to be a sample or set of samples that provides a typical example of general quality of the sampled environmental media in a given area of concern. Sampling locations (specified in the FSP) have been selected to provide detailed chemical information for areas of concern. Chemical parameter analyses were selected based on knowledge of the Site history and results from previous investigations, and will provide adequate chemical characterization of areas of concern.

For the Koppers Pond RI, a combination of semi-random and systematic sampling will be used to obtain representative samples of sediment and surface water to control sampling design error. Sediment and surface water samples from within the pond will be randomly collected as grab samples within defined subareas (i.e., one per acre) to represent spatial variability within the pond. Surface water and sediment samples from the outlets and outlet channel will be grab samples from locations selected using a nonprobablistic approach, based on historical sample locations or other knowledge of the Site. Fish collections will be made throughout the pond, to the extent possible.

Field Representativeness: During the surface water sampling events, dissolved oxygen, pH, ORP, temperature, and specific conductance will be recorded during sampling to facilitate, to the maximum extent possible, the collection of representative surface water characterization samples. Similarly, pH and ORP field measurements will be collected for sediment samples. Appropriate sample handling and equipment decontamination procedures will be followed to ensure that representative samples are collected.

Laboratory Representativeness: The representativeness of laboratory-generated data will be maintained through careful sample-preparation techniques and sample-tracking procedures. Details regarding these procedures can be found in TestAmerica's Laboratory Quality Manual and Laboratory Quality Management Plan and laboratory SOPs provided in electronic format as Attachment B-1.

4.1.7.5 Comparability

Comparability, as referenced in this QAPP, is defined as the similarity of one unit of measure to another unit of measure. Results from chemical analyses associated with the RI and performed either in the laboratory or in the field will be reported in similar units to existing Site data, to the extent possible.

5.0 CUSTODY PROCEDURES

Sample possession and handling will be traced from collection to the final disposition of the sample. "Custody" is maintained if a sample is:

- In the actual possession of an authorized person,
- In view of an authorized person after being in his possession,
- Locked or sealed up after being in possession of an authorized person, and
- In a secure storage room or similar area.

The following subsections describe sample custody procedures for the field, laboratory, and project files.

5.1 FIELD SAMPLE CUSTODY PROCEDURES

Field chain-of-custody is necessary to maintain and document sample possession prior to and during shipping. The principal documents used to identify samples and document possession are chain-of-custody records (Attachment B-2).

Sample custody will begin when samples are collected. Each sample will be labeled with the following information: unique sample identification number, sample location, date and time of collection, and analyses to be performed. Specific procedures for sample identification and numbering are presented in the FSP. The labeled sample will be placed into an iced cooler in the possession of a sampler. A temperature check container will be included in each shipment.

Sampling personnel are responsible for initiating the chain-of-custody record and maintaining custody of samples until they are relinquished to another custodian or to the shipper. A line item on the field chain-of-custody record will be immediately filled out and initialed by sampling personnel. When all line items are completed, or when the samples are prepared for final packaging before shipment, sampling personnel will sign, date, and write the time on the form. Each individual who handles a sample and who subsequently assumes responsibility for the sample will sign the chain-of-custody form.

Sample containers will be packaged appropriately to prevent breakage during shipment and placed in a cooler. Chain-of-custody forms and any other required sample documentation will be enclosed in a waterproof plastic bag and placed in the cooler or hand-delivered to the laboratory courier. Each cooler will be securely taped shut with strapping tape. Custody seals will then be placed on the front and back of each cooler to detect unauthorized tampering with the samples before receipt by the laboratory. Field chain-of-custody procedures end when the laboratory receives the samples.

5.2 LABORATORY SAMPLE CUSTODY PROCEDURES

After receiving samples shipped from the Site, the project laboratory will maintain a custody record throughout sample preparation and analysis. Laboratory custody procedures for project samples undergoing analyses are specified in the laboratory SOPs.

Project samples will be stored at the laboratory for a period of time related to the type and nature of the samples. When storage times have expired, the laboratory will dispose of the samples in accordance with applicable regulations.

5.3 PROJECT FILES

The Group will be responsible for maintaining original documents in a designated secured area. Copies of field chain-of-custody forms and laboratory reports will be maintained in the Cummings/Riter project file located in Pittsburgh, Pennsylvania. Upon completion of the project, records will be transferred to a document repository established by the Group to be maintained for the duration specified in the Administrative Settlement Agreement (minimum ten years). These files will consist of reports, correspondence, field notes, photographs, logbooks, field calibration data, field analytical data, laboratory data, data usability summary reports, data validation reports, and data assessment reports.

6.0 SAMPLING PROCEDURES

Sample collection procedures and sample locations are described in the FSP. The Group will notify USEPA at least 14 days in advance of sample collection activities.

7.0 CALIBRATION PROCEDURES AND FREQUENCY

Both laboratory and field equipment must be calibrated on a regular basis to ensure the accuracy of analyses. The following subsections outline the procedures and frequency for equipment calibration for this project.

7.1 LABORATORY EQUIPMENT

The project laboratory will conduct chemical analyses on samples collected at the Site. The laboratory is required to follow equipment calibration procedures specified in the appropriate analytical methods specified in Table B2-4 for organic parameters and Table B2-5 for other parameters. The analytical methods specify the procedures and frequencies for initial and continuing calibrations and for evaluating calibration data.

7.2 FIELD EQUIPMENT

Field measurements will be made during surface water and sediment sampling and will include dissolved oxygen, pH, ORP, temperature, and specific conductance. Surface and ground water levels will also be measured as described in the FSP. Table B7-1 lists the minimum calibration frequency for the measured field parameters that can be calibrated. Calibration procedures for the field instruments are specified in the SOPs in the FSP. The echo sounder will be calibrated by using a portable depth gauge at two locations and adjusting the echo sounder to equate to the manual reading.

For most instruments, calibrations will be performed each sampling day. If the results of a calibration do not meet field QC acceptance criteria for accuracy, the instrument response will be adjusted to agree with the calibration standard, using the calibration procedures specified in the FSP SOPs. If acceptable calibration cannot be obtained, the associated data will be flagged "J" to indicate the data are estimated.

Calibration standards used by Cummings/Riter on this project will be either directly traceable to the National Institute of Standards and Technology or commercially prepared standards of certified accuracy. Lot numbers of commercially prepared standards will be recorded.

8.0 ANALYTICAL PROCEDURES

Analytical procedures are selected to meet the often conflicting requirements of sensitivity (low detection limit), specificity (correct chemical identification), and speed (interval between sampling and availability of results). A combination of field and laboratory analytical procedures will be followed during the RI at the Site.

The project laboratory (TestAmerica) will perform the analytical testing. TestAmerica has a documented quality system complying with *EPA Requirements for Quality Management Plans for Environmental Data Operations (QA/R-5)* (March 2001). The TestAmerica Laboratory Quality Manual and Laboratory Quality Management Plan are provided as Attachment B-1. TestAmerica Pittsburgh does not participate in the CLP; thus, if requested by USEPA, performance evaluation samples will be analyzed to demonstrate the capability to conduct the required analyses.

Chemical analyses of surface water, sediment, potential source, and fish samples will be conducted by the laboratory for parameters listed in Table B2-4 for organic parameters and Table B2-5 for remaining parameters. Analytical methods for sediment and water will consist of appropriate SW-846 methods using the TCL organic and TAL inorganic analyte lists. Quantitation limits for each analytical parameter are also provided in Tables B2-4 and B2-5.

Field analyses for surface water samples will include dissolved oxygen, pH, ORP, temperature, and specific conductance. For sediment, field measurements include pH and ORP. Field parameters will be measured with standard commercial equipment. Specific methods are included with the SOPs in the FSP.

9.0 INTERNAL QUALITY CONTROL CHECKS

An internal QC system is a set of routine internal procedures for assuring that the data output of a measurement system meets prescribed criteria for data quality. Inherent and implied in this control function is a parallel function of measuring and defining the quality of the data output. A well-designed internal QC program is capable of controlling and measuring the quality of the data in terms of precision and bias. Precision reflects the influence of the inherent variability in any measurement system. Bias represents a consistent error in the measurement system.

For samples collected at the Site, the internal QC measures described in the following subsections will be applied to ensure a high degree of precision and accuracy.

9.1 FIELD QUALITY CONTROL CHECKS

As a check on field sampling QA/QC, trip blanks, equipment rinsate samples, and field duplicates will be sent to the laboratory at specified frequencies. The frequencies at which these samples will be collected and the numbers of such samples are discussed in the following subsections.

Field QC checks also include regular and continuing calibration of measuring equipment. This equipment includes multi-parameter water quality meters for aqueous samples.

9.1.1 Trip Blanks

A trip blank for liquid samples is a sample bottle filled by the laboratory with analyte-free reagent water, handled like a sample, but not opened, and returned to the laboratory for analysis. Trip blanks are analyzed for VOCs only and are used to determine if VOCs are introduced during sample handling and shipment. One trip blank will be included with each shipping cooler of VOC samples sent to the laboratory.

9.1.2 Equipment Rinsate Blanks

Equipment rinsate samples are defined as analyte-free deionized water poured into or pumped through the sampling device, transferred to the sample bottle, then transported to

the laboratory for analysis. These samples help determine whether dedicated sampling equipment was inadvertently contaminated, or if sampling equipment moved between sampling locations was sufficiently decontaminated so as to prevent cross-contamination between samples. The equipment rinsate blanks will be analyzed for the same parameters as the sampled media. Equipment rinsate blanks will be collected at a frequency of 1 for every 20 samples collected or, if less than 20 samples are collected, then 1 per medium sampled.

9.1.3 Field Duplicate Samples

A field duplicate is defined as two or more samples collected independently at a sampling location during a single act of sampling. Procedures for collecting field duplicate samples are described in Section 5.0 of the FSP.

Field duplicates will be indistinguishable by the laboratory from other samples. Therefore, one complete sample set will be identified with a "coded" or false identifier in the same format as other identifiers used for this sample matrix. Both the coded and the true identifiers will be recorded in the field notebook. On the chain-of-custody forms, the coded identifier will be used. These coded field duplicates are used to assess the representativeness of the sampling procedure as well as laboratory precision. One field duplicate sample will be collected for every 20 samples collected or, if less than 20 samples are collected, then 1 duplicate per medium.

9.2 Laboratory Quality Control Checks

QC data are necessary to determine precision and accuracy of the analyses and to demonstrate the absence of interferences and contamination of glassware and reagents. The SW-846 methods to be followed for this project include the use of laboratory blanks, matrix spikes, initial and continuing calibrations, and similar measures. A sample preparation blank will also be prepared during the filleting and homogenization of the fish tissue in the laboratory.

10.0 DATA REDUCTION, VALIDATION, AND REPORTING

The data reduction, validation, and reporting process includes the steps between the instrument or visual reading and the final complete report. Data reduction includes calculations for unit conversions, dilutions and similar factors, and preparation of the initial report. To validate the data, someone other than the analyst reviews the data reduction procedures to determine the acceptability of the data and any necessary qualifiers. Reporting includes transcribing these validated data into the final report and interpretation of the data. Reduction and validation differ among analytical methods, but the reporting process is common to all data.

10.1 DATA REDUCTION

The project laboratory conducting analyses on environmental samples collected during the RI will be required to follow data reduction procedures specified in the SW-846 and other analytical methods identified in Tables B2-4 and B2-5 for organic parameters and remaining parameters, respectively. The PCB results will be reported as both individual Aroclors and as total PCBs, calculated as the sum of the positively detected Aroclor PCBs. If PAHs are detected, they will be reported as the individual PAHs, low and high molecular weight PAHs (L-PAHs and H-PAHs, respectively) and the sum of the potentially carcinogenic PAHs (C-PAHs) will also be calculated.

Field parameters will be measured by direct observation or by direct reading instruments. Results will be recorded directly on data sheets, and no data reduction is required.

10.2 DATA USABILITY

This section outlines data usability procedures for both laboratory and field measurements.

10.2.1 Laboratory Measurements

A data usability evaluation will be performed on the analytical data in accordance with the procedures listed in the USEPA document, *Guidance on Systematic Planning Using the Data Quality Objectives Process* (February 2006). Third-party data validation will be

conducted by AMEC in accordance with applicable USEPA guidance, including Region II modifications, if any. Approximately 20 percent of laboratory data will undergo full validation with all laboratory records and raw instrument data reviewed. If a problem with the data is discovered then additional validation may be completed.

10.2.2 Field Measurements

Field data will be generated by qualified field personnel and immediately entered on the proper form or in a general field logbook. These data will be regularly reviewed for completeness, consistency, and proper procedures (such as calibration) by the Project Supervisor. If discrepancies are found, the appropriate corrective action, usually a re-measurement will be taken promptly.

Calibration results will be checked to verify that initial and continuing calibrations meet the QC acceptance criteria for accuracy in Table B4-1 and to determine that recalibration and reanalysis of samples occurred when these criteria were not met. Results of duplicate samples will be checked to verify that QC acceptance criteria for precision were met. Field equipment blank results will also be reviewed as a check on equipment decontamination procedures and false-positive results.

10.3 REPORTING

For all of the organic and inorganic analytes based on the SW-846 methods, the laboratory data deliverable package will be comparable to that used for CLP data packages, replacing the CLP-specific control limits with those for SW-846 and/or laboratory-established control limits. Field parameters for surface water samples (dissolved oxygen, pH, ORP, temperature, and specific conductance) and for sediment (pH and ORP) will be recorded on sample collection forms.

Data generated in the field will be initially stored in a project file maintained by the Project Supervisor. As soon as practicable, the file will be transferred to the Cummings/Riter Pittsburgh office and grouped with off-Site laboratory reports and other data into the main project file. This file will be organized to allow ready identification and retrieval of desired information.

Quantitative information will be entered into databases. If manually entered, databases will be printed out, checked against the original data sheets, and corrected before use. Cummings/Riter will then use existing programs (and any necessary modifications) to produce data appendices. Any modified programs used to manipulate data will be tested before use with an actual or known data set. Completed data appendices will be checked against the original data sheets.

11.0 PERFORMANCE AND SYSTEMS AUDITS

Laboratory and fieldwork conducted as part of the Koppers Pond RI/FS project may be subject to performance and systems audits. Performance audits check the operation of a specific study component such as a sampling method or an analytical procedure. Systems audits are broader and include a thorough evaluation of both laboratory and field QA methods, such as data validation procedures, corrective action procedures, or sample custody procedures. Audits may be internal (conducted by QA personnel within the organization being audited) or external (conducted by USEPA or another outside agency).

Audits are randomly scheduled by QA personnel and are generally not announced beforehand. If QA personnel find what seems to be a systematic problem with a particular component of the sampling and analysis program, they will normally perform a series of audits on related activities to identify and correct the problem. Audit results are incorporated into the project reporting system, normally in the monthly report.

11.1 LABORATORY AUDITS

If requested by USEPA, the Group will conduct an independent audit of the project laboratory to verify analytical capability and compliance with the SAP. The audit will be conducted sometime during laboratory analyses of project samples. The project laboratory participates in the National Environmental Laboratory Accreditation Program (NELAP).

11.2 FIELD AUDITS

Internal performance and systems audits of field activities at the Site will be coordinated by the QA Officer. A field audit will be conducted at the request of USEPA to verify that project sampling procedures are being correctly followed.

A checklist will be prepared based on information contained in the QAPP, FSP, and HASP. Using the checklist, auditors will evaluate whether field personnel are operating in compliance with procedures specified in these plans, including:

- Initial and continuing equipment calibration;
- Field measurements;
- Sample collection;
- Sample labeling, handling, and custody;
- Data collection and recordkeeping;
- Equipment and personnel decontamination; and
- Health and safety monitoring.

Audit reports will be submitted to USEPA within 15 days of completion of the audit. The report will summarize audit findings, including series deficiencies that adversely reflect the data. Any corrective action taken will also be included in the report.

12.0 PREVENTATIVE MAINTENANCE

Preventative maintenance (PM) includes inspecting, repairing, and adjusting equipment and instruments before deficiencies have a significant effect on performance. These techniques are a necessary part of the procedures for carrying out a particular operation with a particular type of equipment.

12.1 LABORATORY EQUIPMENT

The project laboratory will follow necessary PM actions detailed in its internal SOPs as well as PM required by the analytical methods. These include 1) tuning and calibration (both initial and continuing) of machines, 2) use of internal standards, and 3) related activities such as corrective action. Details of these requirements are included in the methods and the TestAmerica Laboratory Quality Management Plan (Attachment B-1).

12.2 FIELD EQUIPMENT

Cummings/Riter will perform regular PM of field equipment. Field monitoring equipment will be maintained and calibrated in accordance with the manufacturers' recommended schedules and procedures. Field personnel will maintain records of service, calibration, and use. Instrument problems encountered in the field will be detailed in the field daily log and dealt with on Site, if possible.

The primary PM technique for field instruments is the preliminary calibration of equipment. This typically includes a battery check, zero adjustment, and a linearity (or high end) adjustment. Some special items, such as keeping the pH electrode tip wet and refilling it with electrolyte, are required for specific equipment. Failure to calibrate or maintain calibration during an analysis requires corrective action, as discussed in Section 14.2.

To minimize down time in the field, Cummings/Riter maintains an inventory of backup instruments and commonly stocks spare parts for field equipment. Spare parts and backup equipment can be shipped to any field site within 24 hours of request. Typical spare parts for these instruments include D-cell batteries, replacement probes, and

maintenance kits (including O-rings and gaskets) included with the instrument. Cummings/Riter also maintains agreements with instrument rental companies to ensure availability of backup instruments.

13.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The QA objectives described in Section 4.0 are the goals the Group believes are necessary to satisfactorily complete RI field activities at the Site. This section discusses the means for assessing whether objectives have been met. The assessment is a part of the data-handling process described in Section 10.0. Tables B2-4 and B2-5 provide analytical methods, quantitation limits associated with these methods, and data precision and accuracy objectives for organic and remaining analytical parameters, respectively.

13.1 LABORATORY RESULTS

The precision of laboratory results will be determined primarily by calculating the Relative Percent Difference (RPD) for duplicate samples. These will include field duplicates, laboratory duplicates, and MS/MSD samples. The laboratory will determine the accuracy of results by calculating percent recovery values for surrogates and MS/MSD samples. In addition, the laboratory will use laboratory blanks, calibration standards, and internal standards to establish analytical accuracy, as detailed in the methods. Completeness of laboratory results will be determined by comparing the number of validated, usable results to the number of samples planned.

13.2 FIELD RESULTS

The precision of field measurement results will be determined by the use of replicate measures. Accuracy of field results will be determined by evaluating instrument response to suitable standards, such as purchased standard solutions for pH. Completeness for field data will be determined by comparing the number of acceptable measurements with the number specified in the FSP.

13.3 CALCULATIONS

The primary statistic used for estimating precision is RPD for duplicate measurements. RPD is calculated as follows:

$$RPD = \frac{|X_1 - X_2|}{(X_1 + X_2)/2} \times 100$$

where X_1 and X_2 are the results of duplicate measurements and $|X_1-X_2|$ is the absolute value of the difference in the two measurements.

If there are three or more replicates, the percent relative standard deviation (% RSD) will be calculated as a measure of precision:

$$\%RSD = (SD / \overline{X}) \times 100$$

where $\overline{X}$ is the average of the data points (X_1, X_2, . . . X_n) and SD is the standard deviation of the individual measurements.

Accuracy can be estimated by calculating the percent difference (%D) between an instrument response and a known standard:

$$\%D = (S - X) / S \times 100$$

where S is the concentration of a known standard and X is the measured instrument response. This determination of accuracy can be used for both laboratory and field measurements.

Alternatively, accuracy can be measured as percent recovery (%R) from the analytical results of surrogate or analyte compounds spiked into a sample:

$$\%R = (M - N) / S \times 100$$

where M is the measured analyte concentration in the spiked sample, N is the concentration of the analyte in the original sample, and S is the analyte concentration spiked into the original sample. This measurement of accuracy is most appropriate for laboratory results.

Percent completeness (%C) is a measure of 1) the number of samples actually collected compared to the number of samples required for characterization and 2) the amount of valid data obtained compared to the amount of data expected under normal conditions. For the RI, the "number of samples required for characterization" and the "amount of data expected under normal conditions" is the same as the number of samples planned, N. Thus, percent completeness can be defined as:

$$\%C = V / N \times 100$$

where V is the number of valid results and N is the total number of samples planned.

Percent completeness can also be measured as the percent of samples planned that were actually collected:

$$\%C = C / N \times 100$$

where C is the number of samples collected and N is the total number of samples planned. Percent completeness will be calculated on an analytical chemical class basis (e.g., %C for VOCs, %C for SVOCs).

14.0 CORRECTIVE ACTION

Corrective action will be initiated whenever statistical measures indicate exceedance of a control unit. These situations may be identified during performance or system audits or by the analysts/samplers themselves. Corrective action may take place in the laboratory or in the field.

14.1 LABORATORY CORRECTIVE ACTION

If QC audits identify a noncompliance, the problem will be reported to the USEPA. Frequently, problems with analyses result from matrix effects, which make results questionable (estimates, qualified as "J") or unusable (rejected, qualified as "R"). The laboratory and the QA Officer will jointly determine the acceptability of data and the appropriate corrective action. Corrective actions may include:

- Reanalyzing samples if holding time criteria permit,
- Resampling and analyzing the samples,
- Evaluating and amending sampling and analytical procedures, and
- Accepting data and acknowledging a level of uncertainty.

14.2 FIELD CORRECTIVE ACTION

Field analyses will be conducted for aqueous samples. Corrective actions for problems with field analyses will usually be resolved within Cummings/Riter, with occasional input from USEPA or the analytical laboratory. A typical instance would be a pH meter that fails the battery check. The operator will put in a new battery or recharge it and resume calibration. A total failure of an instrument can usually be resolved by sending another instrument to the Site by overnight courier and repeating the analyses the next day.

During field investigations, problems that affect the collection of samples and monitoring data will be documented and recorded in a field log by the person who identified the problem. Serious problems that affect overall project objectives will be brought to the attention of the Project Manager. The Project Manager will notify the Project Coordinator. The Project Manager, Project Supervisor, or their designees are responsible for identifying the causes of the problems and developing a solution.

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TABLES

TABLE B2-1

COPCs FOR SEDIMENT

Area of Concern	Pesticides/PCBs	Inorganics
Koppers Pond	Endrin ketone, Endrin (total), Endosulfan II, 4,4'-DDD, 4,4'-DDT, *Alpha* -chlordane, *Gamma*-chlordane, Aroclor 1254	Aluminum, antimony, barium, beryllium, cadmium, chromium, cobalt, copper, lead, mercury, nickel, silver, vanadium, zinc, cyanide.
Outlet Streams	*Delta* -BHC, Heptachlor, Heptachlor epoxide, 4,4'-DDT, Endrin aldehyde, *Gamma*-chlordane	Aluminum, antimony, barium, beryllium, cadmium, chromium, cobalt, copper, lead, nickel, selenium, silver, thallium, vanadium, zinc

COPCs FOR SURFACE WATER

Area of Concern	Pesticides/PCBs	Inorganics
Koppers Pond	Alpha-BHC Beta-BHC, Aroclor 1254	Aluminum, antimony, arsenic, barium, cadmium, calcium, chromium, copper, iron, lead, magnesium, manganese, potassium, sodium, zinc, and fluoride.

Notes:
1. COPCs for sediment were taken from Table 2-4 in CDM (February 1999).
2. Sediment COPC screening was based only on comparisons of sediment results to screening benchmarks.
3. Sediment COPCs from the Industrial Drainageway were excluded from this compilation considering that sediments from this drainageway were removed in 2003.
4. These COPCs are based on historical data. The RI sampling will generate current data and update COPCs for the Risk Assessment.

TABLE B2-2

SUMMARY OF SAMPLING PROGRAM

SAMPLE MATRIX	FIELD PARAMETERS	LABORATORY PARAMETERS[a]	SAMPLES	FIELD DUPLICATES	MS/MSD SAMPLES	EQUIPMENT RINSATE BLANKS[b]	TRIP BLANKS[c]
Surface Water and Seeps	Oxidation/ Reduction Potential, pH, Temp., Specific Conductance, Dissolved Oxygen	Full TCL/TAL Plus ammonia, fluoride, hardness, nitrites, and total suspended solids	13[d]	1[d]	1[d]	1[d]	1[d]
Sediment[e]	pH, Oxidation/ Reduction Potential	Full TCL/TAL, and total organic carbon	17-44	2	2	2	2
Fish Tissue	--	TCL PCBs/Pesticides, TAL, lipid content[f]	12-24	--	1-2	--	--
Pipe Floc	--	Full TCL/TAL	1	--	--	--	--
Barrier Well Treated Discharge/ Cutler-Hammer Discharge/Chemung Street Outfall	Oxidation/ Reduction Potential, pH, Temp., Specific Conductance, Dissolved Oxygen	Full TCL/TAL Plus ammonia, fluoride, hardness, nitrites, and total suspended solids	1 (each potential source)	--	--	--	--

(a) Parameters include: Full TCL/TAL includes VOCs, SVOCs, pesticides/PCBs, and TAL inorganics. TAL inorganics analyses of aqueous samples will include both the dissolved and total fractions.

(b) Equipment rinsate blanks will not be collected if disposable sampling tools are used.

(c) One trip blank will be shipped with each container submitted to the laboratory for VOC analyses. The total number of trip blanks in the table is an estimate.

(d) Samples to be analyzed sequentially. The numbers of actual QC samples will be prorated according to the actual number of field samples.

(e) Select sediment samples (up to six) will be analyzed for grain size analysis and for acid volatile sulfide/simultaneously extracted metals (AVS/SEM). The locations selected for grain-size and AVS/SEM analyses may or may not correspond.

(f) If, upon field inspection collected fish specimens show deformities that may be indicative of PAH impacts, selected fish tissue samples will also be analyzed for TCL SVOCs.

TABLE B2-3

SAMPLE CONTAINERS, VOLUMES, PRESERVATIVES, AND HOLDING TIMES

PARAMETER	CONTAINER	CONTAINER VOLUME	NO. OF CONTAINERS	PRESERVATIVES	HOLDING TIME [1]
Water:					
TCL VOCs	glass	40 ml septa	3	HCl	10 days
TCL SVOCs	glass	1000 ml	2	Ice	7 days to extract; 40 days to analyze extract
TCL Pesticides/PCBs	glass	1000 ml	3	Ice	7 days to extract; 40 days to analyze extract
TAL Inorganics (total)	plastic	500 ml	1	HNO_3	6 months except for Hg 26 days
TAL Inorganics (dissolved fraction)	plastic	500 ml	1	HNO_3 if field filtered Ice if lab filtered	6 months except for Hg 26 days
Cyanide	plastic	250 ml	1	NaOH	14 days
Hardness	plastic	500	1	HNO_3	28 days
Fluoride, Nitrites (expressed as N), TSS	plastic	1000 ml	1	Ice	48 hours (Nitrites)
Ammonia	plastic	250 ml	1	H_2SO_4	28 Days
Sediments:					
TCL VOCs	glass	4 oz.	3	Ice	10 days
TCL SVOCs, Pesticides/PCBs	glass	8 oz.	3	Ice	14 days to extract; 40 days to analyze extract
TAL Inorganics	glass	8 oz.	1	Ice	6 months except for Hg 26 days
Total Organic Carbon	glass	4 oz.	1	Ice	14 days
Acid Volatile Sulfides/ Simultaneously Extracted Metals	glass	4 oz.	3	Ice	14 days to extract; 28 days to analyze extract
Fish Tissue:					
TCL Pesticides/PCBs and Lipid Content[a] TAL Inorganics	plastic bag	Whole Fish (wrapped in hexane-rinsed aluminum foil)	1	Ice	14 days to extract from thaw; 40 days to analyze extract 6 months except for Hg 26 days
Floc					
TCL VOCs	glass	4 oz.	3	Ice	10 days
TCL SVOCs, TCL Pesticides/PCBs	glass	8 oz.	3	Ice	5 days to extract; 40 days to analyze extract
TAL Inorganics	glass	8 oz.	1	Ice	6 months except for Hg 26 days

(a) If upon field inspection, collected fish specimens show deformities that may be indicative of PAH impacts, selected fish tissue samples will also be analyzed for TCL SVOCs.

Parameter	Analytical Method		Quantitation Limit		
	Water/ Sediments	Fish Tissue	Water (ug/l)	Sediment (ug/kg)	Tissue (ug/kg)
Volatiles		–			
Acetone	SW 846 8260B	–	5	20	–
Benzene	SW 846 8260B	–	1	5	–
Bromodichloromethane	SW 846 8260B	–	1	5	–
Bromoform	SW 846 8260B	–	1	5	–
Bromomethane	SW 846 8260B	–	1	5	–
2-Butanone	SW 846 8260B	–	5	5	–
Carbon disulfide	SW 846 8260B	–	1	5	–
Carbon tetrachloride	SW 846 8260B	–	1	5	–
Chlorobenzene	SW 846 8260B	–	1	5	–
Dibromochloromethane	SW 846 8260B	–	1	5	–
Chloroethane	SW 846 8260B	–	1	5	–
Chloroform	SW 846 8260B	–	1	5	–
Chloromethane	SW 846 8260B	–	1	5	–
Cyclohexane	SW 846 8260B	–	1	5	–
1,2-Dibromo-3-chloropropane	SW 846 8260B	–	1	5	–
1,2-Dibromoethane	SW 846 8260B	–	1	5	–
1,2-Dichlorobenzene	SW 846 8260B	–	1	5	–
1,3-Dichlorobenzene	SW 846 8260B	–	1	5	–
1,4-Dichlorobenzene	SW 846 8260B	–	1	5	–
Dichlorodifluoromethane	SW 846 8260B	–	1	5	–
1,1-Dichloroethane	SW 846 8260B	–	1	5	–
1,2-Dichloroethane	SW 846 8260B	–	1	5	–
cis-1,2-Dichloroethene	SW 846 8260B	–	1	5	–
trans-1,2-Dichloroethene	SW 846 8260B	–	1	5	–
1,1-Dichloroethene	SW 846 8260B	–	1	5	–
1,2-Dichloropropane	SW 846 8260B	–	1	5	–
cis-1,3-Dichloropropene	SW 846 8260B	–	1	5	–
trans-1,3-Dichloropropene	SW 846 8260B	–	1	5	–
Ethylbenzene	SW 846 8260B	–	1	5	–
2-Hexanone	SW 846 8260B	–	5	5	–
Isopropylbenzene	SW 846 8260B	–	1	5	–
Methyl acetate	SW 846 8260B	–	1	5	–
Methylcyclohexane	SW 846 8260B	–	1	5	–
Methylene chloride	SW 846 8260B	–	1	5	–
4-Methyl-2-pentanone	SW 846 8260B	–	5	5	–
Methyl tert-butyl ether	SW 846 8260B	–	1	5	–
Styrene	SW 846 8260B	–	1	5	–
1,1,2,2-Tetrachloroethane	SW 846 8260B	–	1	5	–
Tetrachloroethene	SW 846 8260B	–	1	5	–
Toluene	SW 846 8260B	–	1	5	–
1,2,4-Trichlorobenzene	SW 846 8260B	–	1	5	–
1,1,1-Trichloroethane	SW 846 8260B	–	1	5	–
1,1,2-Trichloroethane	SW 846 8260B	–	1	5	–
Trichloroethene	SW 846 8260B	–	1	5	–
Trichlorofluoromethane	SW 846 8260B	–	1	5	–
1,1,2-Trichloro-1,2,2-trifluoroethane	SW 846 8260B	–	1	5	–
Vinyl chloride	SW 846 8260B	–	1	5	–
Xylenes (total)	SW 846 8260B	–	3	15	–
Semi-Volatiles		–			
Acenaphthene	SW 846 8270C LL	–	0.2	6.7	–
Acenaphthylene	SW 846 8270C LL	–	0.2	6.7	–

Parameter	Analytical Method		Quantitation Limit		
	Water/ Sediments	Fish Tissue	Water (ug/l)	Sediment (ug/kg)	Tissue (ug/kg)
Acetophenone	SW 846 8270C LL	--	1	33	--
Anthracene	SW 846 8270C LL	--	0.2	6.7	--
Atrazine	SW 846 8270C LL	--	1	33	--
Benzaldehyde	SW 846 8270C LL	--	1	33	--
Benzo(a)anthracene	SW 846 8270C LL	--	0.2	6.7	--
Benzo(b)fluoranthene	SW 846 8270C LL	--	0.2	6.7	--
Benzo(k)fluoranthene	SW 846 8270C LL	--	0.2	6.7	--
Benzo(ghi)perylene	SW 846 8270C LL	--	0.2	6.7	--
Benzo(a)pyrene	SW 846 8270C LL	--	0.2	6.7	--
1,1'-Biphenyl	SW 846 8270C LL	--	1	33	--
bis(2-Chloroethoxy)methane	SW 846 8270C LL	--	1	33	--
bis(2-Chloroethyl) ether	SW 846 8270C LL	--	0.2	6.7	--
bis(2-Ethylhexyl) phthalate	SW 846 8270C LL	--	1	33	--
4-Bromophenyl phenyl ether	SW 846 8270C LL	--	1	33	--
Butyl benzyl phthalate	SW 846 8270C LL	--	1	33	--
Caprolactam	SW 846 8270C LL	--	1	33	--
Carbazole	SW 846 8270C LL	--	0.2	6.7	--
4-Chloroaniline	SW 846 8270C LL	--	1	33	--
4-Chloro-3-methylphenol	SW 846 8270C LL	--	1	33	--
2-Chloronaphthalene	SW 846 8270C LL	--	0.2	6.7	--
2-Chlorophenol	SW 846 8270C LL	--	1	33	--
4-Chlorophenyl phenyl ether	SW 846 8270C LL	--	1	33	--
Chrysene	SW 846 8270C LL	--	0.2	6.7	--
Dibenz(a,h)anthracene	SW 846 8270C LL	--	0.2	6.7	--
Dibenzofuran	SW 846 8270C LL	--	1	33	--
Di-n-butyl phthalate	SW 846 8270C LL	--	1	33	--
3,3'-Dichlorobenzidine	SW 846 8270C LL	--	1	33	--
2,4-Dichlorophenol	SW 846 8270C LL	--	0.2	6.7	--
Diethyl phthalate	SW 846 8270C LL	--	1	33	--
2,4-Dimethylphenol	SW 846 8270C LL	--	1	33	--
Dimethyl phthalate	SW 846 8270C LL	--	1	33	--
4,6-Dinitro-2-methylphenol	SW 846 8270C LL	--	5	170	--
2,4-Dinitrophenol	SW 846 8270C LL	--	5	170	--
2,4-Dinitrotoluene	SW 846 8270C LL	--	1	33	--
2,6-Dinitrotoluene	SW 846 8270C LL	--	1	33	--
Di-n-octyl phthalate	SW 846 8270C LL	--	1	33	--
Fluoranthene	SW 846 8270C LL	--	0.2	6.7	--
Fluorene	SW 846 8270C LL	--	0.2	6.7	--
Hexachlorobenzene	SW 846 8270C LL	--	0.2	6.7	--
Hexachlorobutadiene	SW 846 8270C LL	--	0.2	6.7	--
Hexachlorocyclopentadiene	SW 846 8270C LL	--	1	33	--
Hexachloroethane	SW 846 8270C LL	--	1	33	--
Indeno(1,2,3-cd)pyrene	SW 846 8270C LL	--	0.2	6.7	--
Isophorone	SW 846 8270C LL	--	1	33	--
2-Methylnaphthalene	SW 846 8270C LL	--	0.2	6.7	--
2-Methylphenol	SW 846 8270C LL	--	1	33	--
4-Methylphenol	SW 846 8270C LL	--	1	33	--
Naphthalene	SW 846 8270C LL	--	0.2	6.7	--
2-Nitroaniline	SW 846 8270C LL	--	5	170	--
3-Nitroaniline	SW 846 8270C LL	--	5	170	--
4-Nitroaniline	SW 846 8270C LL	--	5	170	--
Nitrobenzene	SW 846 8270C LL	--	0.2	6.7	--

Parameter	Analytical Method		Quantitation Limit		
	Water/ Sediments	Fish Tissue	Water (ug/l)	Sediment (ug/kg)	Tissue (ug/kg)
2-Nitrophenol	SW 846 8270C LL	–	1	33	–
4-Nitrophenol	SW 846 8270C LL	–	5	170	–
N-Nitrosodiphenylamine	SW 846 8270C LL	–	0.2	6.7	–
N-Nitrosodi-n-propylamine	SW 846 8270C LL	–	0.2	6.7	–
2,2'-oxybis(1-Chloropropane)	SW 846 8270C LL	–	0.2	6.7	–
Pentachlorophenol	SW 846 8270C LL	–	1	33	–
Phenanthrene	SW 846 8270C LL	–	0.2	6.7	–
Phenol	SW 846 8270C LL	–	0.2	6.7	–
Pyrene	SW 846 8270C LL	–	0.2	6.7	–
2,4,5-Trichlorophenol	SW 846 8270C LL	–	1	33	–
2,4,6-Trichlorophenol	SW 846 8270C LL	–	1	33	–
Pesticides/PCBs					
Aldrin	SW846 8081A	SW846 8081A	0.05	1.7	1.7
alpha-BHC	SW846 8081A	SW846 8081A	0.05	1.7	1.7
beta-BHC	SW846 8081A	SW846 8081A	0.05	1.7	1.7
delta-BHC	SW846 8081A	SW846 8081A	0.05	1.7	1.7
gamma-BHC (Lindane)	SW846 8081A	SW846 8081A	0.05	1.7	1.7
alpha-Chlordane	SW846 8081A	SW846 8081A	0.05	1.7	1.7
gamma-Chlordane	SW846 8081A	SW846 8081A	0.05	1.7	1.7
4,4'-DDD	SW846 8081A	SW846 8081A	0.1	1.7	1.7
4,4'-DDE	SW846 8081A	SW846 8081A	0.1	1.7	1.7
4,4'-DDT	SW846 8081A	SW846 8081A	0.1	1.7	1.7
Dieldrin	SW846 8081A	SW846 8081A	0.1	1.7	1.7
Endosulfan I	SW846 8081A	SW846 8081A	0.05	1.7	1.7
Endosulfan II	SW846 8081A	SW846 8081A	0.1	1.7	1.7
Endosulfan sulfate	SW846 8081A	SW846 8081A	0.1	1.7	1.7
Endrin	SW846 8081A	SW846 8081A	0.1	1.7	1.7
Endrin aldehyde	SW846 8081A	SW846 8081A	0.1	1.7	1.7
Endrin ketone	SW846 8081A	SW846 8081A	0.1	1.7	1.7
Heptachlor	SW846 8081A	SW846 8081A	0.05	1.7	1.7
Heptachlor epoxide	SW846 8081A	SW846 8081A	0.05	1.7	1.7
Methoxychlor	SW846 8081A	SW846 8081A	0.5	3.3	3.3
Toxaphene	SW846 8081A	SW846 8081A	5	67	67
Aroclor 1016	SW846 8082	SW846 8082	0.4	16.67	16.67
Aroclor 1221	SW846 8082	SW846 8082	0.4	16.67	16.67
Aroclor 1232	SW846 8082	SW846 8082	0.4	16.67	16.67
Aroclor 1242	SW846 8082	SW846 8082	0.4	16.67	16.67
Aroclor 1248	SW846 8082	SW846 8082	0.4	16.67	16.67
Aroclor 1254	SW846 8082	SW846 8082	0.4	16.67	16.67
Aroclor 1260	SW846 8082	SW846 8082	0.4	16.67	16.67
Other					
Lipids	–	Lab-Specific	–	–	1000000

Notes:
1. LCS = Laboratory Control Sample
2. MS/MSD = Matrix Spike/Matrix Spike Duplicate

Parameter	QC Limits (%)																	
	Sediment						Water						Fish					
	LCS[1]			MS/MSD2			LCS			MS/MSD			LCS			MS/MSD		
	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy
	Lower limit	Upper Limit		Lower limit	Upper Limit		Lower limit	Upper Limit		Lower limit	Upper Limit		Lower limit	Upper limit		Lower limit	Upper limit	
Volatiles																		
Acetone	10	175	74	10	192	94	10	161	82	10	161	82	–	–	–	–	–	–
Benzene	77	120	20	49	141	38	80	120	32	73	120	32	–	–	–	–	–	–
Bromodichloromethane	70	125	21	66	128	32	66	130	35	64	130	35	–	–	–	–	–	–
Bromoform	57	138	29	45	148	39	46	153	39	46	153	39	–	–	–	–	–	–
Bromomethane	25	154	40	22	171	51	33	171	54	33	171	54	–	–	–	–	–	–
2-Butanone	21	159	71	10	177	84	21	142	64	17	149	64	–	–	–	–	–	–
Carbon disulfide	44	142	26	38	162	37	54	132	48	52	134	48	–	–	–	–	–	–
Carbon tetrachloride	65	128	34	57	134	37	55	150	38	53	150	38	–	–	–	–	–	–
Chlorobenzene	79	120	20	47	146	32	80	120	29	80	120	29	–	–	–	–	–	–
Dibromochloromethane	70	132	23	61	140	32	60	141	37	57	141	37	–	–	–	–	–	–
Chloroethane	22	158	54	15	179	62	23	186	72	23	186	72	–	–	–	–	–	–
Chloroform	72	120	25	67	127	29	72	127	37	67	129	37	–	–	–	–	–	–
Chloromethane	39	140	39	39	155	34	50	139	42	47	139	42	–	–	–	–	–	–
Cyclohexane	64	133	30	47	147	38	50	150	40	50	150	40	–	–	–	–	–	–
1,2-Dibromo-3-chloropropane	36	143	72	27	156	72	37	133	52	35	143	52	–	–	–	–	–	–
1,2-Dibromoethane	69	128	24	63	133	35	74	123	35	68	128	35	–	–	–	–	–	–
1,2-Dichlorobenzene	71	124	22	64	137	28	77	120	24	74	120	24	–	–	–	–	–	–
1,3-Dichlorobenzene	71	125	20	65	137	28	76	120	24	73	120	24	–	–	–	–	–	–
1,4-Dichlorobenzene	70	124	21	61	138	29	77	120	24	74	120	24	–	–	–	–	–	–
Dichlorodifluoromethane	11	157	63	10	175	49	13	174	58	12	174	58	–	–	–	–	–	–
1,1-Dichloroethane	66	124	23	63	133	31	73	126	38	68	130	38	–	–	–	–	–	–
1,2-Dichloroethane	61	127	23	52	135	34	68	132	32	62	137	32	–	–	–	–	–	–
cis-1,2-Dichloroethene	71	122	32	57	136	31	63	128	41	56	136	41	–	–	–	–	–	–
trans-1,2-Dichloroethene	66	122	24	61	132	36	73	126	41	70	128	41	–	–	–	–	–	–
1,1-Dichloroethene	59	129	25	46	143	36	65	136	48	60	139	48	–	–	–	–	–	–
1,2-Dichloropropane	72	122	20	69	129	30	76	124	34	71	124	34	–	–	–	–	–	–
cis-1,3-Dichloropropene	66	126	22	60	132	34	66	120	35	62	120	35	–	–	–	–	–	–
trans-1,3-Dichloropropene	66	132	22	63	135	31	65	125	42	60	129	42	–	–	–	–	–	–
Ethylbenzene	78	125	21	57	145	33	72	126	33	71	127	33	–	–	–	–	–	–
2-Hexanone	35	151	43	27	163	55	25	132	61	18	145	61	–	–	–	–	–	–
Isopropylbenzene	70	133	22	67	140	33	58	130	35	58	130	35	–	–	–	–	–	–
Methyl acetate	27	142	66	20	151	84	50	150	40	50	150	40	–	–	–	–	–	–
Methylcyclohexane	66	135	23	33	160	41	50	150	40	50	150	40	–	–	–	–	–	–
Methylene chloride	58	127	28	47	137	38	63	129	51	59	131	51	–	–	–	–	–	–
4-Methyl-2-pentanone	48	143	32	42	152	45	37	132	55	33	143	55	–	–	–	–	–	–
Methyl tert-butyl ether	48	132	36	44	138	45	64	123	41	56	131	41	–	–	–	–	–	–
Styrene	74	126	22	68	137	29	71	127	34	71	127	34	–	–	–	–	–	–
1,1,2,2-Tetrachloroethane	57	135	26	50	144	39	62	125	35	59	128	35	–	–	–	–	–	–
Tetrachloroethene	74	130	23	65	139	33	70	135	37	67	136	37	–	–	–	–	–	–
Toluene	78	124	21	32	161	54	80	123	35	75	126	35	–	–	–	–	–	–
1,2,4-Trichlorobenzene	51	136	54	42	148	54	49	124	42	49	131	42	–	–	–	–	–	–
1,1,1-Trichloroethane	67	126	31	54	137	35	63	133	37	59	135	37	–	–	–	–	–	–
1,1,2-Trichloroethane	70	128	22	65	135	34	77	127	36	71	131	36	–	–	–	–	–	–
Trichloroethene	76	119	21	46	141	35	73	120	36	53	135	36	–	–	–	–	–	–
Trichlorofluoromethane	10	192	98	16	173	76	51	156	52	43	162	52	–	–	–	–	–	–
1,1,2-Trichloro-1,2,2-trifluoroethane	55	130	37	49	142	44	39	162	57	38	162	57	–	–	–	–	–	–
Vinyl chloride	53	131	36	54	141	33	53	138	45	47	142	45	–	–	–	–	–	–
Xylenes (total)	76	125	21	56	146	33	72	128	32	72	129	32	–	–	–	–	–	–
Semi-Volatiles																		
Acenaphthene	34	107	36	34	107	36	35	96	41	35	96	41	–	–	–	–	–	–
Acenaphthylene	32	122	40	32	122	40	38	105	40	38	105	40	–	–	–	–	–	–

CUMMINGS RITER

Parameter	QC Limits (%)																	
	Sediment						Water						Fish					
	LCS[1]			MS/MSD2			LCS			MS/MSD			LCS			MS/MSD		
	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy
	Lower limit	Upper Limit		Lower limit	Upper Limit		Lower limit	Upper Limit		Lower limit	Upper Limit		Lower limit	Upper limit		Lower limit	Upper limit	
Acetophenone	0	0	0	0	0	0	0	0	0	0	0	0	–	–	–	–	–	–
Anthracene	32	106	35	32	106	35	35	95	37	35	95	37	–	–	–	–	–	–
Atrazine	0	0	0	0	0	0	0	0	0	0	0	0	–	–	–	–	–	–
Benzaldehyde	0	0	0	0	0	0	0	0	0	0	0	0	–	–	–	–	–	–
Benzo(a)anthracene	34	108	32	34	108	32	38	96	36	38	96	36	–	–	–	–	–	–
Benzo(b)fluoranthene	24	113	28	24	113	28	34	99	46	34	99	46	–	–	–	–	–	–
Benzo(k)fluoranthene	28	120	47	28	120	47	32	109	31	32	109	31	–	–	–	–	–	–
Benzo(ghi)perylene	21	131	21	21	131	21	27	116	44	27	116	44	–	–	–	–	–	–
Benzo(a)pyrene	32	122	31	32	122	31	37	112	40	37	112	40	–	–	–	–	–	–
1,1'-Biphenyl	0	0	0	0	0	0	0	0	0	0	0	0	–	–	–	–	–	–
bis(2-Chloroethoxy)methane	40	102	35	40	102	35	39	91	46	39	91	46	–	–	–	–	–	–
bis(2-Chloroethyl) ether	46	95	33	46	95	33	39	92	38	39	92	38	–	–	–	–	–	–
bis(2-Ethylhexyl) phthalate	34	115	31	34	115	31	33	110	40	33	110	40	–	–	–	–	–	–
4-Bromophenyl phenyl ether	37	105	20	37	105	20	39	94	40	39	94	40	–	–	–	–	–	–
Butyl benzyl phthalate	35	110	34	35	110	34	33	106	40	33	106	40	–	–	–	–	–	–
Caprolactam	0	0	0	0	0	0	0	0	0	0	0	0	–	–	–	–	–	–
Carbazole	35	105	38	35	105	38	36	95	35	36	95	35	–	–	–	–	–	–
4-Chloroaniline	26	96	28	26	96	28	5	99	39	5	99	39	–	–	–	–	–	–
4-Chloro-3-methylphenol	37	114	31	37	114	31	41	99	42	41	99	42	–	–	–	–	–	–
2-Chloronaphthalene	34	105	44	34	105	44	34	95	39	34	95	39	–	–	–	–	–	–
2-Chlorophenol	45	99	40	45	99	40	39	93	39	39	93	39	–	–	–	–	–	–
4-Chlorophenyl phenyl ether	35	110	44	35	110	44	37	96	38	37	96	38	–	–	–	–	–	–
Chrysene	35	103	31	35	103	31	36	97	42	36	97	42	–	–	–	–	–	–
Dibenz(a,h)anthracene	22	135	40	22	135	40	29	119	44	29	119	44	–	–	–	–	–	–
Dibenzofuran	33	105	37	33	105	37	36	94	39	36	94	39	–	–	–	–	–	–
Di-n-butyl phthalate	37	108	34	37	108	34	38	99	38	38	99	38	–	–	–	–	–	–
3,3'-Dichlorobenzidine	21	88	30	21	88	30	10	89	56	10	89	56	–	–	–	–	–	–
2,4-Dichlorophenol	39	111	27	39	111	27	41	96	41	41	96	41	–	–	–	–	–	–
Diethyl phthalate	32	112	38	32	112	38	36	103	39	36	103	39	–	–	–	–	–	–
2,4-Dimethylphenol	33	109	42	33	109	42	41	93	40	41	93	40	–	–	–	–	–	–
Dimethyl phthalate	35	111	41	35	111	41	39	100	42	39	100	42	–	–	–	–	–	–
4,6-Dinitro-2-methylphenol	38	129	35	38	129	35	28	128	41	28	128	41	–	–	–	–	–	–
2,4-Dinitrophenol	19	140	43	19	140	43	20	142	53	20	142	53	–	–	–	–	–	–
2,4-Dinitrotoluene	42	118	33	42	118	33	37	120	39	37	120	39	–	–	–	–	–	–
2,6-Dinitrotoluene	39	124	30	39	124	30	40	117	40	40	117	40	–	–	–	–	–	–
Di-n-octyl phthalate	24	141	43	24	141	43	28	125	44	28	125	44	–	–	–	–	–	–
Fluoranthene	35	103	23	35	103	23	36	96	39	36	96	39	–	–	–	–	–	–
Fluorene	33	111	47	33	111	47	36	97	40	36	97	40	–	–	–	–	–	–
Hexachlorobenzene	35	102	29	35	102	29	40	88	35	40	88	35	–	–	–	–	–	–
Hexachlorobutadiene	38	112	25	38	112	25	38	98	41	38	98	41	–	–	–	–	–	–
Hexachlorocyclopentadiene	29	133	33	29	133	33	36	115	47	36	115	47	–	–	–	–	–	–
Hexachloroethane	40	102	37	40	102	37	38	91	39	38	91	39	–	–	–	–	–	–
Indeno(1,2,3-cd)pyrene	18	140	37	18	140	37	32	116	54	32	116	54	–	–	–	–	–	–
Isophorone	36	121	33	36	121	33	44	99	43	44	99	43	–	–	–	–	–	–
2-Methylnaphthalene	33	110	34	33	110	34	38	90	42	38	90	42	–	–	–	–	–	–
2-Methylphenol	40	110	37	40	110	37	43	90	38	43	90	38	–	–	–	–	–	–
4-Methylphenol	40	113	42	40	113	42	41	92	41	41	92	41	–	–	–	–	–	–
Naphthalene	38	103	25	38	103	25	40	89	43	40	89	43	–	–	–	–	–	–
2-Nitroaniline	34	119	34	34	119	34	35	110	65	35	110	65	–	–	–	–	–	–
3-Nitroaniline	32	106	27	32	106	27	11	104	48	11	104	48	–	–	–	–	–	–
4-Nitroaniline	34	113	31	34	113	31	26	107	45	26	107	45	–	–	–	–	–	–
Nitrobenzene	27	127	36	27	127	36	40	99	42	40	99	42	–	–	–	–	–	–

Parameter	QC Limits (%)																	
	Sediment						Water						Fish					
	LCS[1]			MS/MSD2			LCS			MS/MSD			LCS			MS/MSD		
	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy
	Lower limit	Upper Limit		Lower limit	Upper Limit		Lower limit	Upper Limit		Lower limit	Upper Limit		Lower limit	Upper limit		Lower limit	Upper limit	
2-Nitrophenol	49	110	30	49	110	30	43	105	41	43	105	41	–	–	–	–	–	–
4-Nitrophenol	24	132	37	24	132	37	39	110	42	39	110	42	–	–	–	–	–	–
N-Nitrosodiphenylamine	37	99	40	37	99	40	35	93	36	35	93	36	–	–	–	–	–	–
N-Nitrosodi-n-propylamine	39	111	32	39	111	32	41	96	43	41	96	43	–	–	–	–	–	–
2,2'-oxybis(1-Chloropropane)	28	114	31	28	114	31	33	98	42	33	98	42	–	–	–	–	–	–
Pentachlorophenol	18	117	37	18	117	37	23	108	42	23	108	42	–	–	–	–	–	–
Phenanthrene	35	95	20	35	95	20	37	86	36	37	86	36	–	–	–	–	–	–
Phenol	44	100	40	44	100	40	38	95	39	38	95	39	–	–	–	–	–	–
Pyrene	28	116	28	28	116	28	30	106	42	30	106	42	–	–	–	–	–	–
2,4,5-Trichlorophenol	36	113	35	36	113	35	38	98	39	38	98	39	–	–	–	–	–	–
2,4,6-Trichlorophenol	37	116	47	37	116	47	41	97	39	41	97	39	–	–	–	–	–	–
Pesticides/PCBs																		
Aldrin	75	123	12	40	135	37	69	121	22	59	116	50	39	122	40	33	122	40
alpha-BHC	59	127	10	11	133	52	46	133	20	36	131	55	33	130	40	30	130	40
beta-BHC	76	138	14	10	165	47	71	129	24	48	137	39	51	110	43	18	116	43
delta-BHC	20	124	11	10	119	227	10	137	26	10	164	78	19	142	34	16	142	34
gamma-BHC (Lindane)	66	124	10	11	129	75	63	123	21	11	142	49	47	130	36	33	130	36
alpha-Chlordane	71	130	13	33	143	47	67	127	21	64	121	44	39	145	65	26	145	65
gamma-Chlordane	68	123	24	20	141	75	65	121	21	59	124	27	33	154	36	31	154	36
4,4'-DDD	86	135	18	10	194	80	76	128	24	69	132	41	39	157	35	19	157	35
4,4'-DDE	79	133	14	10	218	34	74	125	18	74	126	41	26	157	39	49	157	39
4,4'-DDT	61	126	37	10	155	71	62	120	24	10	156	53	35	144	42	23	144	42
Dieldrin	76	123	18	34	138	39	76	119	20	58	126	36	45	128	33	33	133	33
Endosulfan I	73	126	23	55	125	53	72	123	22	63	123	37	24	113	41	17	113	41
Endosulfan II	81	128	33	23	179	118	72	122	21	62	128	41	35	124	27	21	129	27
Endosulfan sulfate	44	140	26	10	189	147	47	129	21	10	172	103	36	139	34	22	139	34
Endrin	77	127	20	37	141	43	70	125	24	58	131	36	47	133	38	33	138	38
Endrin aldehyde	73	122	17	10	136	182	61	116	25	10	225	45	27	130	29	18	153	29
Endrin ketone	80	132	14	17	147	76	73	123	17	51	135	46	49	137	32	34	137	32
Heptachlor	73	128	7	43	133	36	65	127	25	59	118	51	39	126	44	32	128	44
Heptachlor epoxide	69	131	10	63	119	26	71	122	19	62	122	44	46	125	43	33	148	43
Methoxychlor	75	143	26	48	163	60	59	143	27	56	137	39	24	161	41	25	164	41
Toxaphene	30	150	30	30	150	30	30	150	30	30	150	30						
Aroclor 1016	55	117	35	10	183	39	60	110	27	43	112	35	49	122	39	26	144	39
Aroclor 1221	0	0	0	0	0	0	0	0	0	0	0	0						
Aroclor 1232	0	0	0	0	0	0	0	0	0	0	0	0						
Aroclor 1242	0	0	0	0	0	0	0	0	0	0	0	0						
Aroclor 1248	30	150	35	30	150	35	30	150	35	30	150	35						
Aroclor 1254	50	150	35	50	150	35	50	150	35	50	150	35						
Aroclor 1260	54	117	29	25	143	34	60	111	24	44	121	31	51	127	33	37	138	33
Other																		
Lipids	–	–	–	–	–	–	–	–	–	–	–	–	30	150	25	30	150	25

Notes:
1. LCS = Laboratory Control Sample
2. MS/MSD = Matrix Spike/Matrix Sp

CUMMINGS RITER

Parameter	Analytical Method				Quantitation Limit			
	Sediment	Sediment - AVS/SEM[1]	Water	Fish Tissue	Sediment (mg/kg)	Sediment - AVS/SEM (µmoles/gram)	Water (µg/L)	Fish Tissue (mg/kg)
Aluminum	SW846 6020	–	SW846 6020	SW846 6020	3	–	30	3
Antimony	SW846 6020	–	SW846 6020	SW846 6020	0.2	–	2	0.2
Arsenic	SW846 6020	SW846 6010B	SW846 6020	SW846 6020	0.1	0.003337	1	0.1
Barium	SW846 6020	–	SW846 6020	SW846 6020	1	–	10	1
Beryllium	SW846 6020	–	SW846 6020	SW846 6020	0.1	–	1	0.1
Cadmium	SW846 6020	SW846 6010B	SW846 6020	SW846 6020	0.1	0.001112	1	0.1
Calcium	SW846 6020	–	SW846 6020	SW846 6020	10	–	100	10
Chromium	SW846 6020	SW846 6010B	SW846 6020	SW846 6020	0.2	0.002404	2	0.2
Cobalt	SW846 6020	–	SW846 6020	SW846 6020	0.05	–	0.5	0.05
Copper	SW846 6020	SW846 6010B	SW846 6020	SW846 6020	0.2	0.009835	2	0.2
Iron	SW846 6020	–	SW846 6020	SW846 6020	5	–	50	5
Lead	SW846 6020	SW846 6010B	SW846 6020	SW846 6020	0.1	0.0007239	1	0.1
Magnesium	SW846 6020	–	SW846 6020	SW846 6020	10	–	100	10
Manganese	SW846 6020	–	SW846 6020	SW846 6020	0.05	–	0.5	0.05
Nickel	SW846 6020	SW846 6010B	SW846 6020	SW846 6020	0.1	0.01704	1	0.1
Potassium	SW846 6020	–	SW846 6020	SW846 6020	10	–	100	10
Selenium	SW846 6020	–	SW846 6020	SW846 6020	0.5	–	5	0.5
Silver	SW846 6020	SW846 6010B	SW846 6020	SW846 6020	0.1	0.001159	1	0.1
Sodium	SW846 6020	–	SW846 6020	SW846 6020	10	–	100	10
Thallium	SW846 6020	–	SW846 6020	SW846 6020	0.1	–	1	0.1
Vanadium	SW846 6020	–	SW846 6020	SW846 6020	0.1	–	1	0.1
Zinc	SW846 6020	SW846 6010B	SW846 6020	SW846 6020	0.5	0.03823	5	0.5
Mercury	SW846 7471A	SW846 7470A	SW846 7470A	SW846 7471A	0.033	0.00006232	0.2	0.033
Total Cyanide	SW846 9012A	–	SW846 9012A	SW846 9012A	0.5	–	10	0.5
TOC	TOC Lloyd Kahn	–	–	–	500	–	–	–
Fluoride	–	–	EPA 300.0 IC	–	–	–	0.05	–
Nitrite	–	–	EPA 300.0 IC	–	–	–	0.05	–
Total Suspended Solids	–	–	SM 2540D	–	–	–	4	–
Ammonia	–	–	EPA 350.1	–	–	–	0.1	–

CUMMINGS RITER

Parameter	QC Limits (%)											
	Sediment						Sediment - AVS/SEM					
	LCS[2]			MS/MSD[3]			LCS			MS/MSD		
	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy	Precision		Accuracy
	Lower limit	Upper Limit		Lower limit	Upper limit		Lower limit	Upper Limit		Lower limit	Upper limit	
Aluminum	80	120	20	75	125	20	–	–	–	–	–	–
Antimony	80	120	20	75	125	20	–	–	–	–	–	–
Arsenic	80	120	20	75	125	20	80	120	20	75	125	20
Barium	80	120	20	75	125	20	–	–	–	–	–	–
Beryllium	80	120	20	75	125	20	–	–	–	–	–	–
Cadmium	80	120	20	75	125	20	80	120	20	75	125	20
Calcium	80	120	20	75	125	20	–	–	–	–	–	–
Chromium	80	120	20	75	125	20	80	120	20	75	125	20
Cobalt	80	120	20	75	125	20	–	–	–	–	–	–
Copper	80	120	20	75	125	20	80	120	20	75	125	20
Iron	80	120	20	75	125	20	–	–	–	–	–	–
Lead	80	120	20	75	125	20	80	120	20	75	125	20
Magnesium	80	120	20	75	125	20	–	–	–	–	–	–
Manganese	80	120	20	75	125	20	–	–	–	–	–	–
Nickel	80	120	20	75	125	20	80	120	20	75	125	20
Potassium	80	120	20	75	125	20	–	–	–	–	–	–
Selenium	80	120	20	75	125	20	–	–	–	–	–	–
Silver	80	120	20	75	125	20	80	120	20	75	125	20
Sodium	80	120	20	75	125	20	–	–	–	–	–	–
Thallium	80	120	20	75	125	20	–	–	–	–	–	–
Vanadium	80	120	20	75	125	20	–	–	–	–	–	–
Zinc	80	120	20	75	125	20	–	–	–	–	–	–
Mercury	80	120	20	75	125	20	80	120	20	80	120	20
Total Cyanide	38.4	162	50	75	125	20	80	120	20	75	125	20
TOC	75	125	20	75	125	20	–	–	–	–	–	–
Fluoride	–	–	–	–	–	–	–	–	–	–	–	–
Nitrite	–	–	–	–	–	–	–	–	–	–	–	–
Total Suspended Solids	–	–	–	–	–	–	–	–	–	–	–	–
Ammonia	–	–	–	–	–	–	–	–	–	–	–	–

Parameter	QC Limits (%)											
	Water						Fish					
	LCS			MS/MSD			LCS			MS/MSD		
	Precision Lower limit	Precision Upper limit	Accuracy	Precision Lower limit	Precision Upper limit	Accuracy	Precision Lower limit	Precision Upper limit	Accuracy	Precision Lower limit	Precision Upper limit	Accuracy
Aluminum	80	120	20	75	125	20	75	125	20	75	125	20
Antimony	80	120	20	75	125	20	75	125	20	75	125	20
Arsenic	80	120	20	75	125	20	75	125	20	75	125	20
Barium	80	120	20	75	125	20	75	125	20	75	125	20
Beryllium	80	120	20	75	125	20	75	125	20	75	125	20
Cadmium	80	120	20	75	125	20	75	125	20	75	125	20
Calcium	80	120	20	75	125	20	75	125	20	75	125	20
Chromium	80	120	20	75	125	20	75	125	20	75	125	20
Cobalt	80	120	20	75	125	20	75	125	20	75	125	20
Copper	80	120	20	75	125	20	75	125	20	75	125	20
Iron	80	120	20	75	125	20	75	125	20	75	125	20
Lead	80	120	20	75	125	20	75	125	20	75	125	20
Magnesium	80	120	20	75	125	20	75	125	20	75	125	20
Manganese	80	120	20	75	125	20	75	125	20	75	125	20
Nickel	80	120	20	75	125	20	75	125	20	75	125	20
Potassium	80	120	20	75	125	20	75	125	20	75	125	20
Selenium	80	120	20	75	125	20	75	125	20	75	125	20
Silver	80	120	20	75	125	20	75	125	20	75	125	20
Sodium	80	120	20	75	125	20	75	125	20	75	125	20
Thallium	80	120	20	75	125	20	75	125	20	75	125	20
Vanadium	80	120	20	75	125	20	75	125	20	75	125	20
Zinc	80	120	20	75	125	20	75	125	20	75	125	20
Mercury	80	120	20	75	125	20	75	125	20	75	125	20
Total Cyanide	85	115	20	75	125	20	38	162	20	75	125	20
TOC	–	–	–	–	–	–	–	–	–	–	–	–
Fluoride	90	110	20	80	120	20	–	–	–	–	–	–
Nitrite	90	110	20	80	120	20	–	–	–	–	–	–
Total Suspended Solids	80	120	20	0	0	20	–	–	–	–	–	–
Ammonia	90	110	10	90	110	20	–	–	–	–	–	–

Notes:
1. AVS/SEM = Acid Volatile Sulfides/Simultaneously Extracted Metals
1. LCS = Laboratory Control Sample
2. MS/MSD = Matrix Spike/Matrix Spike Duplicate

CUMMINGS RITER

TABLE B3-1

DISTRIBUTION LIST

INDIVIDUAL	ADDRESS	PHONE NUMBER
Isabel Rodrigues USEPA Project Manager	U.S. Environmental Protection Agency Region II 290 Broadway, 20th Floor New York, NY 10007	(212) 637-4248
Leo Brausch Project Coordinator	131 Wedgewood Drive Gibsonia, PA 15044	(724) 444-0377
William Smith Cummings/Riter Project Manager	Cummings/Riter Consultants, Inc. 10 Duff Road Suite 500 Pittsburgh, PA 15235	(412) 241-4500
John Samuelian AMEC Project Manager	AMEC Earth and Environmental, Inc. 15 Franklin Street Portland, ME 04101	(207) 879-4222
Denise Ladebauche QA Officer	AMEC Earth and Environmental, Inc. 15 Franklin Street Portland, ME 04101	(207) 879-4222
Kenneth Bird Coordinator Cummings/Riter Health and Safety	Cummings/Riter Consultants, Inc. 10 Duff Road Suite 500 Pittsburgh, PA 15235	(412) 241-4500

TABLE B4-1

FIELD QC ACCEPTANCE CRITERIA FOR ACCURACY AND PRECISION

PARAMETER	ACCURACY[a]	PRECISION
pH	±0.1 pH unit	±0.3 pH unit
Specific Conductance	±10 percent	±10 percent
Temperature	±1°C	NS[b]
Dissolved Oxygen	±0.1 parts per million	±10 percent
Oxidation Reduction Potential	±10 percent	NS

a. Accuracy measured against a standard of known concentration.
b. NS = Not Specified.

TABLE B7-1

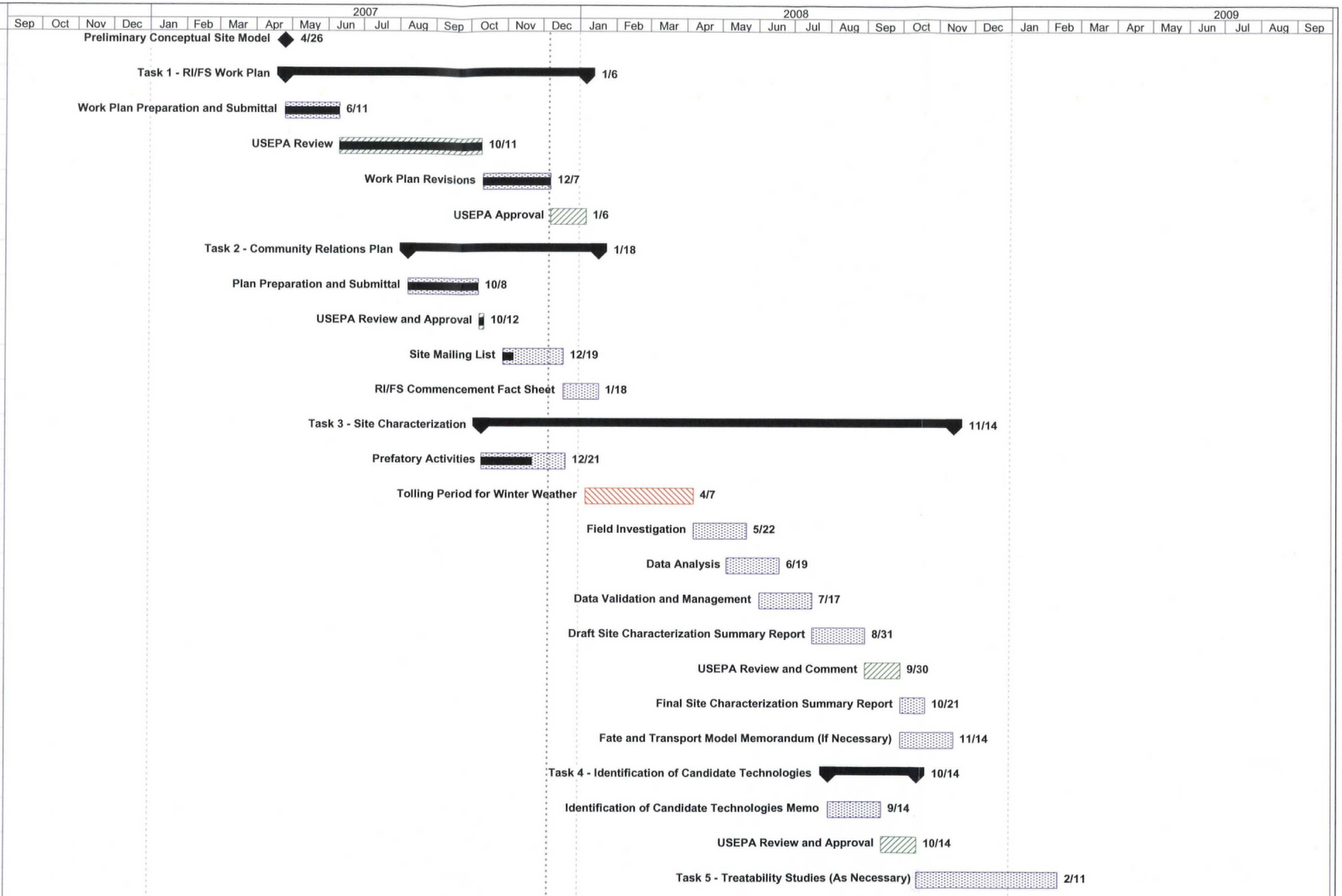
CALIBRATION FREQUENCY FOR FIELD PARAMETERS

PARAMETER	CALIBRATION STANDARDS	CALIBRATION FREQUENCY[a]
pH	pH 4, pH 7, & pH 10	Daily
Oxidation Reduction Potential	231 ± 10 mV	Daily
Dissolved Oxygen	0.0 part per million	Daily
Specific Conductance	100 + 1000 umhos/cm	Daily
Water Level	Measured Steel Tape	Once During Project
Organic Vapors	100 ppm Isobutylene Gas	Daily
Echo Sounder	Measured Steel Tape and Depth Probe	Once During Project

a. Where applicable, instruments will be checked against calibration standards at the beginning of each sampling day (before any field measurements) of the sampling event.

FIGURES

ID	Task Name
1	**Preliminary Conceptual Site Model**
2	**Task 1 - RI/FS Work Plan**
3	Work Plan Preparation and Submittal
4	USEPA Review
5	Work Plan Revisions
6	USEPA Approval
7	**Task 2 - Community Relations Plan**
8	Plan Preparation and Submittal
9	USEPA Review and Approval
10	Site Mailing List
11	RI/FS Commencement Fact Sheet
12	**Task 3 - Site Characterization**
13	Prefatory Activities
14	Tolling Period for Winter Weather
15	Field Investigation
16	Data Analysis
17	Data Validation and Management
18	Draft Site Characterization Summary Report
19	USEPA Review and Comment
20	Final Site Characterization Summary Report
21	Fate and Transport Model Memorandum (If Necessary)
22	**Task 4 - Identification of Candidate Technologies**
23	Identification of Candidate Technologies Memo
24	USEPA Review and Approval
25	**Task 5 - Treatability Studies (As Necessary)**

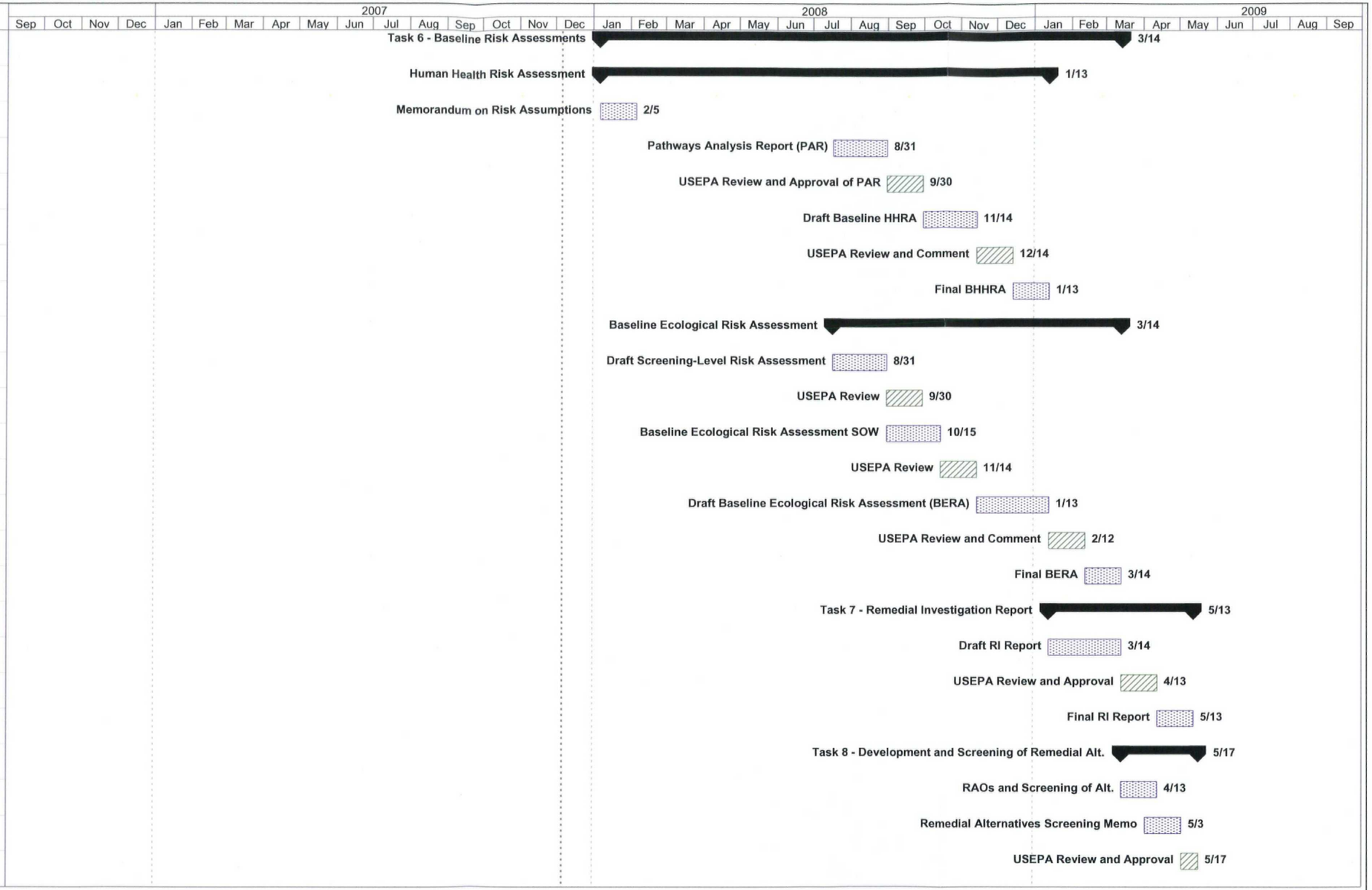


vised Koppers Pond RI/FS Project Schedule
e: Revised RIFS Schedule.mpp
Date: Thu 12/6/07

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks | Group By Summary
Progress | Summary | Rolled Up Milestone | Split | Project Summary | Deadline

FIGURE B1-1, 1 of 3

ID	Task Name
26	**Task 6 - Baseline Risk Assessments**
27	**Human Health Risk Assessment**
28	Memorandum on Risk Assumptions
29	Pathways Analysis Report (PAR)
30	USEPA Review and Approval of PAR
31	Draft Baseline HHRA
32	USEPA Review and Comment
33	Final BHHRA
34	**Baseline Ecological Risk Assessment**
35	Draft Screening-Level Risk Assessment
36	USEPA Review
37	Baseline Ecological Risk Assessment SOW
38	USEPA Review
39	Draft Baseline Ecological Risk Assessment (BERA)
40	USEPA Review and Comment
41	Final BERA
42	**Task 7 - Remedial Investigation Report**
43	Draft RI Report
44	USEPA Review and Approval
45	Final RI Report
46	**Task 8 - Development and Screening of Remedial Alt.**
47	RAOs and Screening of Alt.
48	Remedial Alternatives Screening Memo
49	USEPA Review and Approval

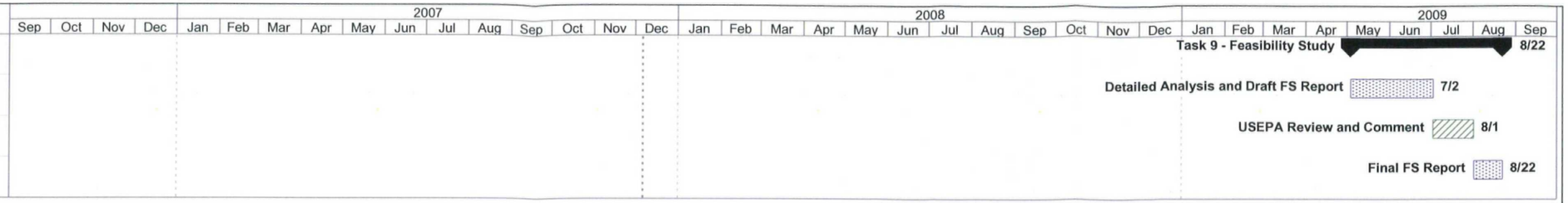


vised Koppers Pond RI/FS Project Schedule
e: Revised RIFS Schedule.mpp
Date: Thu 12/6/07

Task | Milestone | Rolled Up Task | Rolled Up Progress | External Tasks | Group By Summary
Progress | Summary | Rolled Up Milestone | Split | Project Summary | Deadline

FIGURE B1-1, 2 of 3

ID	Task Name
50	**Task 9 - Feasibility Study**
51	Detailed Analysis and Draft FS Report
52	USEPA Review and Comment
53	Final FS Report



ised Koppers Pond RI/FS Project Schedule
: Revised RIFS Schedule.mpp
Date: Thu 12/6/07

Task	Milestone	Rolled Up Task	Rolled Up Progress	External Tasks	Group By Summary
Progress	Summary	Rolled Up Milestone	Split	Project Summary	Deadline

FIGURE B1-1, 3 of 3

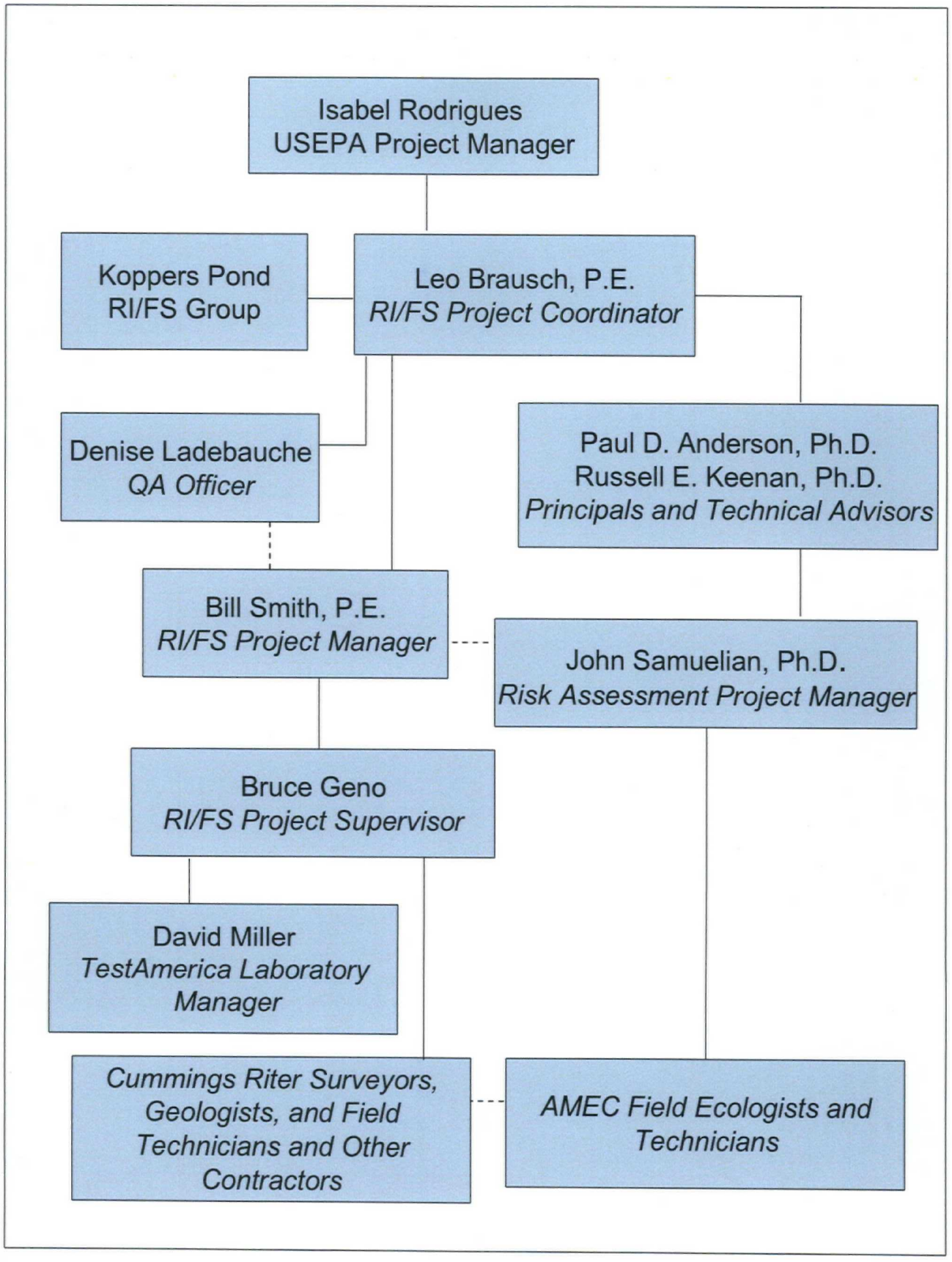


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FIGURE B3-1

PROJECT ORGANIZATION

ATTACHMENT B-1

TESTAMERICA'S LABORATORY QUALITY MANUAL, LABORATORY QUALITY MANAGEMENT PLAN, AND STANDARD OPERATING PROCEDURES

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STL Pittsburgh LQM
Revision No.: 6
Date Revised: January 5, 2007
Implementation Date: Jan 30, 2007

STL Pittsburgh Laboratory Quality Manual

Revision 6

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Purpose and Scope

The purpose of this Laboratory Quality Manual (LQM) is to describe the implementation the Severn Trent Laboratories (STL) Quality System at the STL Pittsburgh laboratory. The LQM is written within the guidelines of the STL Quality Management Plan (QMP), which applies to all STL laboratories. The organization of this LQM is based on the "EPA Requirements for Quality Management Plans" (EPA QA/R-2, August 1994). This LQM outlines specific policies, organization, responsibilities, and activities required to assure high quality laboratory services. The LQM also fulfills the requirements of our clients, government agencies, and NELAC to document the laboratory Quality System.

This LQM contains references to other essential STL quality documents. The company-wide QMP, STL Pittsburgh LQM, and referenced policies and SOPs are interrelated. Together they provide an integrated quality foundation that meets the objectives of the STL Quality Assurance Policy, as stated in Section 1.2.

The requirements set forth in this document are applicable to all employees at the STL Pittsburgh laboratory. The policies and practices described here are presented as minimum guidelines only. Based on good scientific judgment, more rigorous requirements may be applied by laboratory employees. Specific requirements delineated in project plans may supersede general quality requirements described in this manual.

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1.0 Management Commitment and Organization

1.1 STL Mission Statement

We enable our customers to create safe and environmentally favorable polices and practices by leading the market in scientific and consultancy services. We provide this support within a customer service framework that sets the standard to which others aspire. This is achieved by people whose professionalism and development is valued as the key to success and through continued investments in science and technology.

1.2 STL Quality Assurance Policy

It is STL's policy to:

- provide high quality, consistent, and objective environmental testing services that meet all relevant federal, state, and municipal regulatory requirements;
- generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use;
- provide STL clients with the highest level of professionalism and the best service practices in the industry;
- build continuous improvement mechanisms into all laboratory administration, and managerial activities; and
- maintain a working environment that fosters open communication with both clients and staff.

1.3 STL Management Statement of Commitment to Quality Assurance

STL management is committed to providing the highest quality data and the best service in the environmental testing industry. To ensure that the data produced and reported by STL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL maintains a Quality System that is clear, effective, well communicated, and supported at all levels in the company.

1.4 Ethics, Waste, Fraud and Abuse

Establishing and maintaining a high ethical standard is an important element of a Quality System. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times, STL has established an Ethics Policy, P-L-006, and an Ethics Agreement (see Figure 1.4-1). Each employee shall sign the Ethics Agreement , signifying agreed compliance with its sated purpose. A central tenant is that management must consistently convey the message to analysts that financial pressures can never be allowed to compromise the quality of work.

See the following policies are some of the principle documents related to ethics in the laboratory:

- Ethics Policy P-L-006
- Fraud Policy P-L-0007
- Data Recording Requirements Policy QA-008
- Manual Integrations S-Q-004
- Selection of Data Points Required for an Initial Calibration Curve P-T-001

1.5 Organizational Structure and Relationships

STL Pittsburgh is a local operating unit of Severn Trent Laboratories, Inc., a Delaware corporation. Date of incorporation was August 27, 1997. Severn Trent Laboratories is wholly owned by Severn Trent Services, Inc.

The organizational structure for Severn Trent Laboratories, Inc. is presented in QMP Figure 1 and LQM Figure 1.5-1. The responsibilities and authorities of the members of the STL corporate staff employees are described in the STL QMP.

STL Pittsburgh has day-to-day independent operational authority overseen by corporate officers (e.g., President, Commercial Director, Chief Operating Officer, Corporate Quality Assurance, etc.). The STL Pittsburgh laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for STL Pittsburgh is presented in Figure 1.5-2. A list of key STL Pittsburgh personnel qualifications is provided in Figure 1.5-3. The lab also maintains Job Descriptions which contain general job responsibilities for all laboratory employees. The following section outlines responsibilities and authorities for all employees of the STL Pittsburgh laboratory, as they relate to quality management.

The STL Pittsburgh QA Manager (QAM) is independent from day-to-day laboratory operations, has no direct analytical testing responsibilities, and is free from financial and other undue pressures which might adversely affect the quality of work. The QAM, a key member of the laboratory's management team, has direct access to the Corporate Quality Assurance Director on all matters involving quality. The QAM is available to any lab employee to resolve quality or ethical issues. The QAM, if required, has the authority to cease operations adversely affecting the validity or integrity of the analytical data.

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Figure 1.4-1

STL Ethics Agreement

It is the policy of STL to incorporate the highest standard of quality with all analytical programs by adhering to the following practices:

STL will only offer environmental analyses for which it can consistently demonstrate compliance with high quality, traceable and legally defensible performance standards.
All STL staff is committed to the practice of complete honesty in the production and reporting of data.
Staff who are aware of misrepresentation of facts or data manipulation to bypass established QA/QC requirements, are required to immediately inform their supervisor or any member of the upper management.

All employees are asked to sign a copy of the statement below upon their first day of employment.

I, _____(print name) understand that high standards of integrity are required of me with regard to the duties I perform and the data I report in connection with my employment at the Company. I agree that in the performance of my duties at the Company:
I will not intentionally report data values that are not the actual values obtained;
I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;
I will not intentionally misrepresent another individual's work; and
If a supervisor or a member of STL management requests me to engage in or perform an activity that I feel is compromising data validity or quality, I will not comply with the request and report this action immediately to a member of the upper management, up to and including the president of Severn Trent Laboratories Inc.
I will not intentionally report data values that do not meet established quality control criteria as set forth in the Method and/or Standard Operation Procedures, or as defined by Company Policy.

I agree to inform my Supervisor of any accidental reporting of non-authentic data by me in a timely manner. I agree to inform my Supervisor of any accidental or intentional reporting of non-authentic data by other employees. I have read this Ethics Agreement and understand that failure to comply with the conditions stated above will result in disciplinary action, up to and including termination from the Company.

Compliance with this policy of business ethics and conduct is the responsibility of every STL employee. Disregard or failing to comply with this standard of business ethics and conduct could lead to disciplinary action, up to and including possible termination of employment.

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Figure 1.5-1
STL Pittsburgh Organizational Structure

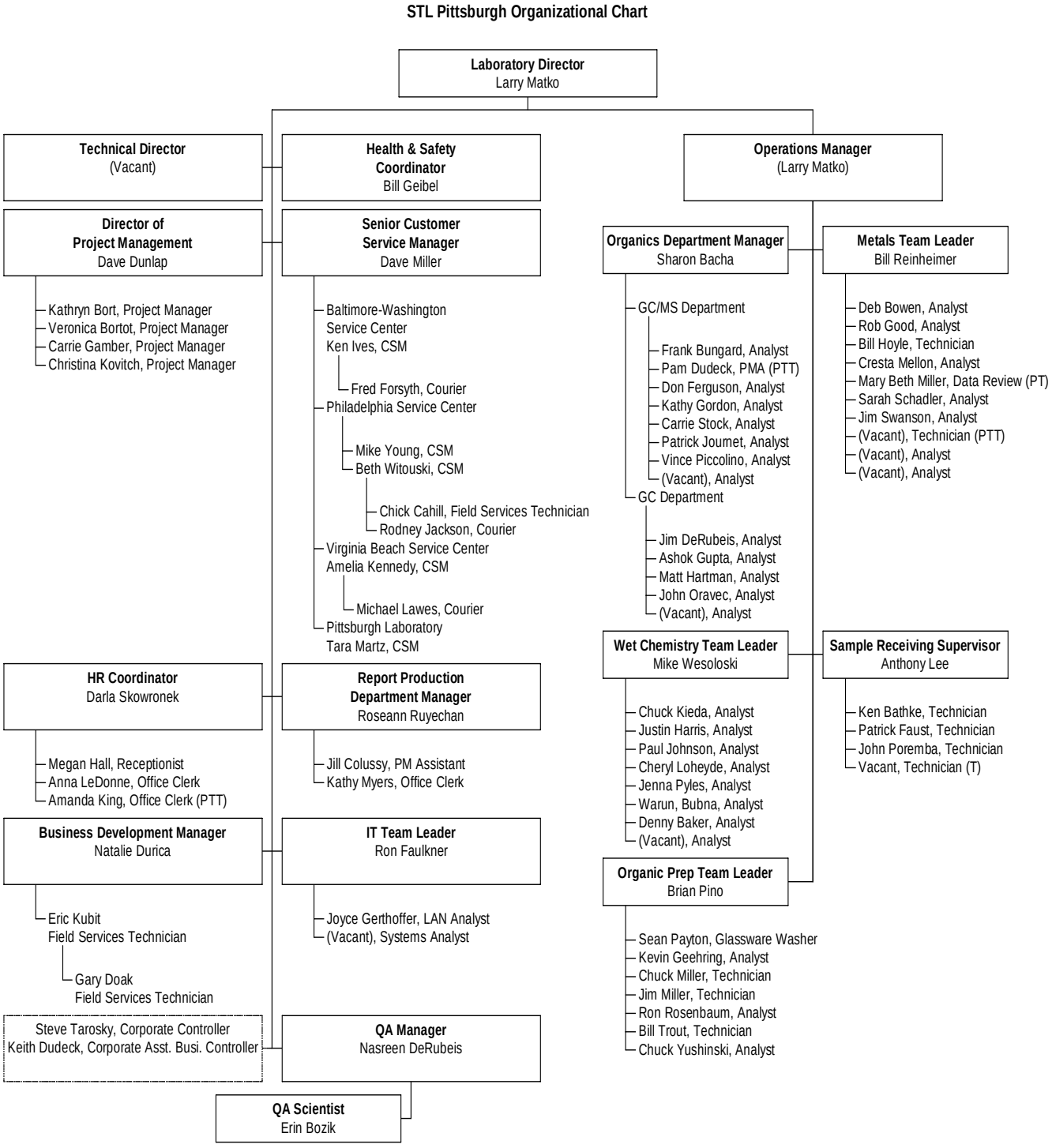

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Figure 1.5-2

STL Pittsburgh Key Personnel

Employee	Title	Degree	Yrs Company (as of 2006)	Yrs Industry (as of 2006)
Vicinie, Albert	General Manager	BS, Chemistry & Biology	10	25
Miller, Dave	Sr. Customer Service Manager	BA, Biology	6	17
Matko, Larry	Lab Director	BS, Chemical Engineering	17	18
DeRubeis, Nasreen	QA Manager	BS, Biology	1	19
Dunlap, David	Director of Project Management	BS, Chemistry	21	21
Bortot, Veronica	Project Manager	BA, Biology	22	22
Gamber, Carrie	Project Manager	BS, Biology	13	19
Kovitch, Christina	Project Manager	AS, Busi Admin	16	16
Bort, Kathy	Project Manager	AS, Lab Techn/ Chemistry	3	24
Bacha, Sharon	Organic Department Manager	MS, Forensic Sci. BA, Chemistry	19	19
Pino, Brian	Supervisor, Organic Prep	NA	12	12
Lee, Anthony	Supervisor, Sample Control	NA	7	26
Reinheimer, Bill	Supervisor, Metals	BS, Chemistry	6	16
Ruyechan, Roseann	Supervisor, Reporting	BS, Biology	19	20
Wesoloski, Mike	Supervisor, Wet Chem	BS. Biology	17	18

1.6 Quality Organization

All personnel are responsible for quality, which includes complying with all QA/QC requirements that pertain to their organizational/technical function. This section outlines the primary QA responsibilities for each position. Detailed job descriptions for all laboratory positions are maintained on the STL Intranet.

1.6.1 Quality Assurance Manager

- Reports directly to the Laboratory Director and, for all QA matters and to the Corporate QA Director to maintain independence from the local operations for which they have quality assurance oversight.
- Responsible for the implementing and communicating the QMP
- Maintains, approves, and implements the LQM
- Has joint signature authority, with the Laboratory Director and Technical Supervisors for approval of quality documents
- Directs controlled distribution laboratory quality documents
- Provides Quality System training to all new personnel
- Reviews and approves documentation of analyst training records
- Serves as a focal point for QA and QC issues, reviews corrective actions and recommends resolution for recurring nonconformances within the laboratory
- Assists in maintaining regulatory analytical compliance, including maintaining certifications, and in this regard has signature authority for laboratory quality documents
- Monitors data quality measures via statistical methods to verify that the laboratory routinely meets stated quality goals
- Performs systems, data, contract compliance, and surveillance audits.
- Hosts external audits conducted by outside agencies
- Responsible for approving quality control reference data changes in the LIMS
- Oversees the selection, review, and approval of analytical subcontractors
- Prepares monthly QA Reports to management describing significant quality events
- Has the final authority to accept or reject data and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data

1.6.2 Laboratory Director

- Reports directory to the General Manager.
- Responsible for implementation and adherence by lab staff to the STL QMP, STL Pittsburgh LQM and all policies and procedures within the laboratory.
- Has signature authority for LQM, policies, SOPs, and contracts (as detailed in STL policy)
- Annually assesses the effectiveness of the QMP and LQM within the operation
- Maintains adequate trained staffing documented on organization charts
- Responsible for implementing internal/external audit findings corrective actions.

1.6.3 Operations Manager

- Reports directly to the Laboratory Director

- Supervises daily activities of the Operational Groups
- Schedules analytical operations
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Supervises maintenance of instruments and scheduling of repairs
- Works with the Project Managers and Group/Team Leaders to assure the requirements of projects are met in a timely manner
- Responsible for meeting laboratory quality requirements

1.6.4 Technical Director

- Reports directly to Laboratory Director
- Responsible for the technical operation of the laboratory
- Responsible for coordinating the development and implementation of SOPs
- Has joint signature authority for LQM, SOPs, and training records
- Performs technical training in area(s) of expertise
- Interfaces with management on technical needs and solving day-to-day technical issues
- Determines qualifications required for technical positions and evaluates job candidates against those requirements
- Investigates technical issues related to projects as directed by QA
- Evaluates new methods, technical proposals, and statements of work
- Certifies technical laboratory personnel based on education and background to ensure that staff have demonstrated capability in the activities for which they are responsible
- Performs other tasks as required by NELAC.
- The Technical Director meets the requirements specified in the Section 4.1.1.1 of the NELAC standards. See Group Leaders for operations specific Technical Supervisors.

1.6.5 Report Production Manager

- Reports directly to the Laboratory Director
- Supervises daily activities of the Report Production Groups
- Works with the Operations Manager and/or Group/Team Leaders to ensure the requirements of projects are met in a timely manner

1.6.6 Customer Service Managers (CSMs)

- Reports directly to the Laboratory Director
- Has signature authority for contracts for laboratory services, as detailed in STL policy, and for laboratory reports.
- Defines customer requirements through project definition
- Assesses and assures customer satisfaction
- Provides feedback to management on changing customer needs

- Brings together resources necessary to ensure customer satisfaction.

1.6.7 Project Manager

- Reports directly to the Director of Project Management.
- Monitors analytical and QA project requirements for a specified project
- Acts as a liaison between the client and the laboratory staff
- Prepares Quality Assurance Summary (QAS) or equivalent summary form and communicates project-specific requirements to all parties involved
- Assists the laboratory staff with interpretation of work plans, contracts, and QAPP requirements
- Reviews project data packages for completeness and compliance to client needs
- Has signature authority for final reports
- Keeps the laboratory and client informed of project status
- Together with the QA Manager, approves customer requested variances to methods and to standard laboratory protocols
- Monitors, reviews, and evaluates the progress and performance of projects
- Reports client inquiries involving data quality issues or data acceptability to the facility QA Manager and to the operations staff
- Prepares reissue requests for project data
- Responsible for meeting quality requirements.

1.6.8 Group Leader or Technical Supervisor

- Reports directly to the Operations Manager.
- Supervises daily activities of analyses within the group
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Evaluates instrument performance and supervises the calibration, preventive maintenance, and scheduling of repairs
- Oversees or performs review and approval of all analytical data
- Reports nonconformances to the appropriate managers
- Responsible for meeting data quality requirements.
- Analytical Group Leaders serve as Technical Directors within their analytical group.
- Responsible for ensuring that all staff within their group are trained and documented as trained to perform the procedures that they are assigned.
- Responsible for generation and maintenance of SOPs for their section
- Responsible for ensuring work done in their area is performed in a compliant manner

1.6.9 Analyst

- Performs analytical methods and data recording in accordance with approved and documented laboratory procedures

- Performs and documents calibration and preventive maintenance
- Performs data processing and data review procedures
- Reports nonconformances to the Supervisor/Manager and QA Manager
- Ensures sample and data integrity by adhering to internal chain-of-custody procedures
- Responsible for meeting quality requirements defined in this LQM and other supporting QA procedures.

1.6.10 Sample Custodian

- Ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody
- Reports nonconformances associated with condition-upon-receipt of samples
- Logs samples into the LIMS
- Ensures that all samples are stored in the proper environment
- Assists Environmental Health and Safety staff with sample disposal
- Responsible for meeting quality requirements.

1.6.11 Report Production Staff

- Accurately generates and compiles analytical reports and associated deliverables for delivery to the client
- Responsible for meeting quality requirements
- Produce as needed reports that meet the NELAC requirements.

2.0 Quality System and Description

2.1 Objective of the STL Quality System

The Quality System is a set of management principles, objectives, policies, responsibilities, and implementation plans at the organizational and project-specific levels. The goal of the STL Quality system is to ensure that business operations are conducted with the highest level of professionalism in the industry. To achieve this goal, it is necessary to provide STL clients with not only scientifically sound, well documented, and regulatory compliant data, but also to ensure that STL provides the highest quality service available in the industry. A well-structured and well-communicated Quality System is essential in meeting this goal. STL's Quality System is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

2.2 Structure of the STL Quality System

At the highest level, the STL Quality Management Plan (QMP) is the basis for STL's Quality System. The QMP provides the guidance under which all STL facilities conduct their operations. This Laboratory Quality Manual (LQM) describes the implementation of the Quality System at the STL Pittsburgh laboratory. This LQM and the series of associated quality documents described in Section 2.4 define the organization, project-specific principles, goals, controls, and tools of the Quality System as it is applied at this laboratory. The Quality System as described in this LQM demonstrates the commitment to accepted laboratory practices by STL Pittsburgh.

2.3 Quality Assurance and Quality Controls

Quality Assurance (QA) is defined as the system of activities which ensures the quality of a process, product, or service. Quality Controls (QC) are the tools used to monitor and regulate the desired type and quality of product. The QA activities and QC controls employed in STL Pittsburgh are defined in the following quality documents.

2.4 Quality Documents

The STL Quality System is developed from the reference documents shown in Table 2.4-1. The review and control of the STL Pittsburgh documents described in the following subsections is described in Section 3 of this LQM. A cross-reference of the LQM to NELAC requirements quality manuals is presented in Table 2.4-2.

2.4.1 STL Quality Management Plan (QMP)

The requirements set forth in the QMP are applicable to all STL facilities. The policies and practices outlined in the QMP are minimum guidelines only. Requirements that are more rigorous may be applied for specific client or regulatory programs.

2.4.2 STL Company-Wide Policies

Severn Trent Laboratories has certain policies that apply company-wide. These policies are consistent with the QMP, and set forth requirements that all STL facilities are to follow. These documents are "controlled" by corporate QA and are posted on the STL intranet for local laboratory access.

2.4.3 Laboratory Quality Manual (LQM)

This STL Pittsburgh LQM along with the associated policies and SOPs, provides the criteria and specifications for the generation of environmental analytical data. The LQM provides QC criteria for standard procedures, facility-specific instrumentation, and reporting.

2.4.4 Standard Operating Procedures

Standard Operating Procedures (SOPs) describe step-by-step instructions for performing a method or activity. In addition, there are SOPs, which relate to other support services performed in the laboratory. Details of SOP format and document control are described in SOP PITT-QA-0010. SOPs that are actively used in this laboratory are listed in Table 8.2-2. SOPs are living documents and may supersede some requirements in this document until the LQM is updated annually.

2.4.5 Quality Assurance Project or Program Plans (QAPPs)

Regulations and contracts may contain QA requirements which are different from those described in this LQM. To address unique project requirements, Quality Assurance Project Plans (QAPjPs) may be prepared and implemented. The requirements documented in a QAPjP, as agreed to by STL Pittsburgh, take precedence over the LQM for that project. Typical specifications contained in a QAPjP or similar documentation include:

- New or modified testing methods
- Unique QC logic
- Special requirements for equipment use and maintenance
- Special handling due to safety considerations
- Project-specific detection and reporting limits
- Project-specific accuracy and precision limits or the statistical treatment of data
- Additional or unique documentation or records management requirements.

2.4.5.1 Quality Assurance Summary

Quality Assurance Summaries (QAS) or equivalent (e.g., Client/ Project Checklist) are used to distill client-specific requirements typically documented in project QA plans onto a concise format, highlighting the requirements that are different than the laboratory standard practice. The summary describes for each project the required quality control samples, batching schemes, flagging conventions, deliverables, or other special client requests that may differ from routine laboratory operations. The QAS or equivalent is disseminated to laboratory operations by the Project Manager or Quality Assurance Manager to document client or program specific requirements. The QAS may be used alone or in conjunction with the project-specific QA plans.

2.4.6 Other Documents

Other documents which can affect the quality program may include the Chemical Hygiene Plan (CHP), memos, guidance documents, work instructions, and periodic management assessment reports. These documents may further define or guide the implementation of quality standards at STL but shall not conflict with the LQM or diminish the effectiveness of the Quality System.

3.0 Document Control and Records Management

3.1 Objectives for Control of Quality Documents and Vital Records

Quality Documents - The quality documents discussed in Section 2 define the framework of the STL Quality System. Control and security of these documents are necessary to ensure that all staff have access to current policies and procedures at all times, to ensure that all changes to the policies and procedures are properly reviewed, to ensure that the history of use of documents can be reconstructed, and to ensure that confidential information is not improperly distributed. The system described in this section is designed to accomplish these objectives.

Vital Records – Vital records are the documents that provide objective evidence of the performance of a process or observations of an item. Records management ensures that results produced by the laboratory are scientifically and legally defensible, and ensures that project events can be reconstructed. Confidentially of the records and records retention requirements are discussed in this section.

3.2 Document Control Procedures

Unambiguous identification of a controlled document is maintained by identification of the following items in the document header:

- document title,
- unique document number,
- revision number,
- revision date,
- effective or implementation date, and number of pages

Controlled documents are marked as such, and the QA department keeps records of document distribution. Controlled distribution may be achieved by either electronic or hardcopy means. The effective date is the date when controlled copies are distributed. Controlled documents are available electronically via a web page. Details of the numbering system, required format, and restrictions for uncontrolled distribution of documents are in SOP PITT-QA-0010, “Tracking, Review and Revision of SOPs” and the STL Policy No. S-Q-001, “Official Document Control and Archive”.

3.3 Document Review, Approval and Revision

Controlled quality documents are authorized by the Laboratory Director, the Technical Manager, and the QA Manager. They indicate their authorization by signing the cover page of the document. STL Pittsburgh quality documents, the individuals responsible for reviewing the documents, and the required frequency of review are listed in Table 2.4-3 and Table 2.4-4. In addition to periodic review and revision, quality documents must be revised when a procedure or activity changes in a significant manner. Amendments to documents must be reviewed and approved by the same parties approving the original document, distributed in a controlled manner, and clearly indicated in the document. Obsolete versions of documents are removed from service when new revisions are issued. The QA Department maintains a record of history of all documents. For further details see SOP PITT-QA-0010, “Tracking, Review and Revision of SOPs”.

3.4 Records Management

Records may be either hardcopy or electronic copies. It is not required to maintain both if they are properly secured and are complete and true copies. The record keeping system allows for reconstruction of all laboratory activities that produced the analytical results. The history of the sample is readily understood through the documentation. This includes

- chain-of-custody records, including intra-laboratory and inter-laboratory transfers of samples;

- records identifying the personnel involved in sampling, preparation, calibration, and testing;
- observations, calculations, and derived data;
- information relating to laboratory facilities, equipment, analytical test methods, and related laboratory activities (e.g., sample preparation, standards preparation, and data verification);
- original records clearly identifying all subcontracted test data, and
- a copy of the final test report.

Requirements for data recording are described in Policy # QA-008, "Data Recording Requirements". Details concerning control of electronic records are given in Section 6. The types of vital records maintained are listed in Table 3.4-1.

3.5 Document and Record Storage, Retention and Disposal

It is the policy of STL Pittsburgh that company records will be available to meet business needs and comply with all applicable legal records retention and disposition requirements. STL Pittsburgh retains copies of records in a manner that allows prompt retrieval of documents and records for inspection purposes. In accordance with NELAC, all quality documents and records are stored for at least five years. Other types of records have different retention requirement, refer to Table 3.4-1 for details.

Specific projects and regulatory programs have longer record retention requirements than the standard STL record retention time. Refer to the STL QMP Table 5 for a listing of examples of special program requirements. The inventory sheet accompanying the stored records must include disposal instructions which take into account any special requirements, and who to contact for authorization prior to destroying the data.

When records, as contained in files, are transferred to a records storage area or off-site storage area, they shall be placed in suitable containers and include an inventory sheet (hard copy or electronic) prepared by the person submitting the records. The contents of each container shall be compared to the inventory sheet and labeled. If there are any discrepancies, the container and inventory sheet shall be returned to the person who prepared the box for correction. Archives are indexed such that records are accessible on a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Backup copies of electronic media are stored in off-site archive facilities and are protected against deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented. Further details of the laboratory's document and records archiving process are described in SOP PITT-QA-0019.

If the laboratory transfers ownership, vital records will be transferred to the new owner. If the laboratory goes out of business, vital records will be transferred to another operating STL laboratory or to our clients.

3.6 Data Confidentiality

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL and client contract) subject to any disclosure required by law or legal process. STL's reports, and the data and information provided therein, are for the exclusive use and benefit of client, and are not released to a third party without written consent from the client. In some cases the client may identify projects requiring confidentiality due to national security. Information concerning these projects will be limited only to those STL Pittsburgh associates with a need to know. The audit reports supplied by federal, state, and local regulatory agencies are public information and can be released without written consent of those agencies. However, specific client audits are confidential and must be approved by the client before releasing them to a third party.

4.0 Staff Qualification, Orientation and Training

All activities performed by STL Pittsburgh shall be accomplished by qualified personnel. Each staff member must have the combination of experience and education needed to demonstrate the required knowledge for his or her position. Each must also have an appropriate general knowledge of laboratory operations, test methods, quality assurance and quality control procedures, and records management. Minimum training requirements are shown in

Figure 4-1 at the end of this Section. SOP C-QA-0013 describes details of the training process and documentation. The Chemical Hygiene Plan (CHP) describes details for health and safety training.

4.1 Qualifications

STL Pittsburgh maintains job descriptions for all positions. These job descriptions specify the minimum qualifications for education and experience, knowledge and skills, which are necessary to perform at a satisfactory level. Qualifications of professional staff are documented by resumes that include academic credentials, employment history, experience, and professional registrations. A copy of each person's resume is maintained in an electronic file, and is readily available for inspection.

4.2 Orientation and Technical Training

Each new staff member shall receive orientation in quality and in health and safety. Each new staff member shall be supervised in their assigned duties by their supervisor or a knowledgeable individual designated by the supervisor. The ability and authorization to perform independently shall be documented in the training files, as described below, with technical duties approved by the Technical Director or designee.

4.2.1 Quality Assurance (QA) Orientation

Each new staff member will receive a QA orientation. The QA Manager or designee will conduct this orientation within two weeks of the new employee's first day on the job. The orientation will, at a minimum, include the following topics:

- STL Quality System and hierarchy of quality documents (QMP, LQM, policies, and SOPs);
- key elements of the LQM and the Quality Control Policy (QA-003);
- introduction to the nonconformance memo (NCM) system and corrective action procedures;
- proper data recording practices;
- STL ethics agreement, including the potential consequences of unethical behavior; and
- the role of the QA department.

The QA orientation will be documented on a checklist, which is signed by the trainee. The documentation will be placed in the employee's training file.

4.2.2 Quality Training

Continued training in the mission and goals of the QMP and LQM shall be provided at least annually. These may be done in a single session or divided into separate sessions conducted at different times throughout the year. Formal training sessions are conducted and documented by the QA Manager or designee. In addition, each lab staff member shall read and document their awareness of the quality documents related to his or her position.

4.2.3 Ethics Training

4.2.3.1 Ethics is also a major component of the STL QA training program. Each employee must be trained in ethics within 30 days of hire in a QA training program that includes an overview of regulatory programs and program goals, a review of the ethics statement, and group discussions about data integrity and data misrepresentation. Employees must be trained as to the legal and environmental repercussions that result from data misrepresentation. A data integrity hotline is maintained by STL and administered by QA Director.

4.2.4 Health and Safety, Orientation and Training

Each new employee, contract worker, or working visitor is required to go through health and safety orientation and training as described in the CHP. The Health and Safety Coordinator must conduct the orientation as soon as possible after the individual reports to work and before chemicals are handled. More comprehensive health and safety training, both initial and on going, must be completed at the frequency given in the CHP.

4.3 Training Files

Each active STL Pittsburgh staff member has an individual training file maintained by the QA Manager or designee. This file can be documented on paper forms or in a database. The following sections shall be included in the training files at a minimum:

- Resume - containing hardcopy or a reference to the electronic file
- Quality Assurance - containing documentation of QA/QC orientation and training completed
- Health and Safety - orientation and training documents
- Technical Proficiency - initial and on-going demonstrations of proficiency, one-on-one training, training courses or workshops on specific equipment or analytical methods is documented in this file. Note that proficiency with technical SOPs is document on the demonstration of capability forms (see next section).

Other types of records to be included in the training file include work place regulatory compliance training, and professional development courses. The exact contents will vary depending upon a person's job function and tenure with the company. Details of requirements for training records and the approval process are given in SOP C-QA-0013.

4.4 Technical Proficiency Demonstrations

All new personnel are required to demonstrate competency in performing a particular method by successfully completing a Demonstration of Capability (DOC) before conducting analysis independently on client samples. On-going proficiency must be demonstrated annually.

DOCs are most commonly performed by analysis of four replicate QC check samples. Results of successive LCS analyses can be used to fulfill the DOC requirement. As required by the referenced method, the accuracy and precision, measured as average recovery and standard deviation (using n-1 population), of the four replicates are calculated and compared to the method limits or against current laboratory limits if multi-laboratory method acceptance limits are not specified. Use of single-blind proficiency samples and other NELAC acceptable proficiency samples are described in SOP C-QA-0013. The DOC Certification documentation must be signed by the Technical Director and the Quality Assurance Manager and filed in the employee's training file (see example in C-QA-0013). The DOC Certification documentation must include a statement that the individual has read, understood, and agreed to perform the most recent version of the test procedure. In procedures such as %Solids, pH, Color, Dissolved oxygen, Ignitability etc., where spiking is not an option and for which quality control samples are not readily available, the proficiency can be demonstrated by analyzing a duplicate sample provided the RPD≤10%.

This is a Controlled Document. When Printed it becomes Uncontrolled.

Figure 4-1

Employee Minimum Training Requirements

Required Training	Time Frame	Employee Type
Ethics Training	Initial orientation within 30 days of hire and annual refresher	All
Environmental Health & Safety	Initial training before start of production work.	All
	Additional training as specified in the CHP	As required
Quality Assurance	Orientation within 2 weeks of hire date	All
	Annual QA program training	All
Technical Proficiency	Initial demonstration prior to unsupervised method performance	Technical staff
	Annual on-going demonstration	Technical staff

5.0 Procurement and Supplies and Services

Controlling the quality of supplies and services is necessary to ensure that STL Pittsburgh provides high quality analytical services to our clients. The STL procurement program requires:

- assurance that purchased items and services meet requirements set by STL Pittsburgh and perform as expected
- definition of the levels of documentation required for applicable technical and administrative procurement functions
- maintenance of records of all suppliers from whom we obtain services or supplies required for our analytical testing
-

5.1 Selection of Vendors

Materials and supplies are purchased from approved vendors. Prospective vendors are selected based upon criteria appropriate to the materials or supplies provided. STL Policy # P-PUG-006 "Procurement and Contracts" details the process used. For national vendors and contracts, the vendor is selected by the STL Procurement Director through a competitive bidding process, strategic business alliance or negotiated vendor partnership. Potential vendors are required to complete a vendor acceptance application and are evaluated on the following criteria, as appropriate:

- the vendor's history of providing identical or similar products that perform satisfactorily in actual use
- the vendor's service record and ability to provide a complete product line and commensurate service
- the vendor's ability to administer inventory at the STL Pittsburgh facility through an inventory management system that will ensure correct stocking levels as well as shelf-life tracking
- objective evaluation of the vendor's current quality records, supported by documentation
- results of audits by STL of the vendor's technical and quality capabilities

Vendors that provide measuring equipment, solvents, chemical standards, instrument service contracts, or subcontracted laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items.

5.2 Controlling Quality of Purchased Items

The quality of equipment, reagents, solvents, chemical standards, gases, and laboratory containers used in analyses must be of known quality so that their effect upon analytical results can be defined. These quality specifications are derived from analytical method requirements, project-specific requirements, and defined national standards for analytical testing. Quality specifications of materials are described in analytical SOPs. These quality specifications shall be included or referenced in the purchasing documents for the items being purchased. This includes specifications for the purity of standards, reagents, or chemicals, and technical specifications for accuracy and precision (e.g., Class A volumetric glassware). Reference to a catalogue number, model, lot number, or chemical grade is sufficient.

Quality materials, reagents, and dry chemicals are verified upon receipt to ensure the suitable grade of material was received. Upon ordering materials, the lab ensures that the item ordered is received and meets requirements.

The Laboratory Director has the responsibility for approving purchase orders. The section supervisors or designees are responsible for ensuring that the requested quality of materials ordered matches those received, for verifying that material storage is properly maintained and for removing materials from use when shelf life has expired.

5.2.1 Evaluation of Off-The-Shelf Items

For items that are used regularly by STL Pittsburgh where no unique requirements or specifications exist, the items may be purchased off-the-shelf. These items are ordered from the supplier on the basis of specifications set forth in the supplier's published product description. These include items such as glassware, filter paper, pipettes, and chromatography columns. The items are evaluated as a function of the standard analytical process.

5.2.2 Evaluation of Instruments

Evaluation of instruments purchased shall be conducted according to an acceptance testing plan. The acceptance testing plan may be defined by the vendor or the method demonstration requirements specified in the laboratory analytical SOPs. Acceptance criteria may include instrument reliability, sensitivity, stability, selectivity, accuracy, precision, and ability to interface with existing computer systems.

5.2.3 Evaluation of Critical Solvents and Acids

STL Pittsburgh is part of a group of STL laboratories that conducts additional evaluations for certain solvents and chemical reagents where our criteria for purity are more stringent than the vendor's. These chemicals are listed in Table 5.2-1. These chemicals are subject to analysis on a lot-by-lot basis before they are put into use. They are tested at one of the STL laboratories, and the chemical test results are evaluated by a designated quality representative. If the solvents or reagents meet the specifications given in S-T-001 The Testing of Solvents and Acids, the lot is released to the laboratory by the approved vendors. All laboratories then use the same lot, and reject any lots received at the facility that have not been tested.

5.2.4 Evaluation of Chemical Standards and Reference Materials

Where available chemical standards will be traceable to the National Institute of Standards Technology (NIST) or an equivalent source. This is largely limited to physical and inorganic chemical standards. If NIST traceability is not commercially available, commercially certified materials shall be used, which are then tested for accuracy before reporting data. Details of the testing procedures and documentation are described in the laboratory SOP. Standards must be received with a certification report from the vendor with information such as purity/concentration, traceability, lot number, expiration date, preparation date, unique identification number, formula weight, density, mass and/or volume of standards, and suggested storage requirements. Further details about labeling and handling of standards is described in Section 8 of this LQM.

5.2.5 Corrective Action for Failure to Meet Required Specifications

Corrective actions for failure of an item to meet required specifications are as follows:

- review of current supplies to eliminate the problem item
- notification to the STL Procurement Director to avoid additional problems at other STL labs
- return of the problem item to the vendor
- evaluate the impact on product or process

The QA Manager shall be notified of any significant or systematic quality problems. The STL Procurement Director and the STL Quality Assurance Director shall be notified of any quality problems with national vendors.

5.3 Procurement of Subcontract Laboratory Services

Whether external to STL or not, all subcontracting from the STL Pittsburgh laboratory to another laboratory is arranged with the documented consent of the client, in a timely response that shall not be unreasonably refused. 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. Documentation of required certifications from the subcontract facility are maintained in STL project records. Where applicable, specific QC guidelines, QAPjPs, and similar project documents are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC).

Subcontract laboratories may receive an on-site audit by a representative of STL's QA staff if it is deemed appropriate by the QA Manager. The audit involves an assessment of compliance with the required test method, QC requirements, documentation, as well as any special client requirements.

For DoD projects the subcontractor laboratories used must have an established and documented laboratory quality system that complies with DoD QSM requirements. The subcontractor laboratories will be evaluated according to SOP PITT-QA-0023, Selection and Evaluation of Subcontractor Laboratories. The subcontractor laboratory must receive project-specific approval from the DoD client before any samples are analyzed as per DoD QSM, Version 3.0, Section 4.5.

Project reports received from external laboratories are not altered and are included in original form in the final report provided by STL. Intracompany subcontracting may also occur between STL facilities. The originating laboratory is responsible for communicating QA/QC, reporting, and other project requirements.

The final report from STL Pittsburgh clearly identifies what testing was performed by other laboratories, and, per NELAC, the certification status of the lab performing the work.

6.0 Computer Hardware and Software

The primary purpose of quality assurance systems for computer hardware and software is to protect the integrity of computer-resident data. Procedures are in place at STL Pittsburgh to assure that computer-resident data are accurate, traceable to a known source, protected against loss, and secure.

STL's computer and hardware controls are based on the guidance in EPA's "Good Automated Laboratory Practices" (GALP), August, 1995. This includes both corporate level Information Technology (IT) functions and STL Pittsburgh IT functions. Some GALP requirements, such as management responsibilities and the training program, are addressed in other sections of the LQM. Some corporate level IT functions, such as the system change management procedures, are described in more detail in corporate IT documents. Table 6-1 provides a cross reference of practices outlined in Section 8 of the GALP manual to corresponding sections of STL's QA and IT documents.

6.1 Computer Hardware

Computer hardware used in the generation, measurement, or assessment of client data shall be of appropriate design and adequate capacity to function according to specifications. Computer equipment must be installed in accordance with the manufacturer's recommendations, and undergo documented acceptance testing.

6.1.1 Wide-Area Systems

STL Pittsburgh's LIMS (QuantIMS) and the Office Network run on a wide-area network (WAN) serving multiple laboratories. The central node for the network is located at the Denver facility. The central processor is an IBM AS-400 with multiple servers and Cisco routers. Records for the system architecture, testing and maintenance, such as Initial Program Loads (IPLs), are documented in the AS-400 System Log, which is also in Denver. Records for installation of the network hardware are maintained by the central System Administrator.

6.1.2 Local Systems

The local systems consist of computer equipment for analytical instruments, data evaluation, and upload to the LIMS. A local-area network (LAN) supports the local office software. Testing, maintenance, and repair of the local computer hardware is the responsibility of the STL Pittsburgh LAN Analyst. Documentation for the local systems is maintained by the LAN Analyst.

6.2 Facilities and Security

6.2.1 Central Computer Facilities

The environmental conditions of the facility housing the LIMS are controlled to protect against data loss. Access to the central computer facility in Denver is restricted by keypad entry used by IT staff. The central computer room is temperature controlled, and has an Uninterrupted Power Supply (UPS) plus a power generator to ensure that the WAN functions are not disrupted by power failures. Backup media, such as tapes and disks, are maintained daily.

In addition, full volume backup copies of the raw data are shipped offsite to a commercial facility specially designed to store electronic data.

6.2.2 Local Computer Facilities

Facilities for housing local computer hardware must meet manufacturer's recommendations. Electronic data must be protected against environmental hazards such as fire, water damage, and strong electromagnetic fields. Data files will have backup copies made at regular intervals to protect against accidental loss through hardware or software failure.

6.2.3 Controlled Software Access

The integrity of data is also assured by maintaining limited access to administrative functions through a hierarchy of operating system shells controlled by passwords. Access is granted by the LAN Administrator depending on a persons experience, training, and assigned duties (see SOP S-ITQ-005 for more details).

Firewalls are in place to protect against unauthorized access from the Internet.

6.2.4 Virus Protection

Commercial virus protection programs are installed on all computers to detect and remove computer viruses. LAN Analysts are to be notified whenever a virus is detected so that they can isolate any portions of the systems that may be at risk.

6.3 LIMS Raw Data

QuantIMS raw data and instrument raw data from instrument data systems such as Target, IDB, and Chemstation are stored on the Office Automation servers (e.g., PITSRV0X). The Systems Administrator and the LAN Analyst are responsible for maintaining the servers.

The individuals responsible for entering and recording raw data must be uniquely identified in the data, together with the date and time the data were entered (QA-008 Data Recording). The instrument transmitting raw data must be uniquely identified, together with the date and time of the transmission. Further data recording requirements exist to document manual integrations (see Policy # S-Q-004 for details).

Procedures for verifying raw data are discussed in (QA-012 Technical Data Review) and in LQM Sections 8.8 to 8.8.3.

6.4 Software

If computer software is used to acquire, process, or report client data, that software is tested to ensure that it correctly performs its intended function. Software are validated or verified, depending upon its complexity, size, and whether it was purchased or developed by STL. The following definitions are used by STL:

- Validation - the process of establishing documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting predetermined specifications and quality attributes. This process demonstrates and documents that the software performs correctly and meets all specified requirements.
- Verification - the process of checking the accuracy of automatically (electronically) calculated information.

6.4.1 Industry Standard Software

Industry standard software programs are defined as those which are purchased and widely used without modification to the program itself. The program is initially verified for use by using test problems with known solutions to demonstrate that the program is operational for the desired application.

All purchased software must be used in accordance with the terms of its software license. Any use of software contrary to its license terms is expressly prohibited by STL.

6.4.2 Testing of STL-Developed Software

For programs used to process client data and developed within STL, and externally prepared programs which are modified by STL, validation or verification must be performed. The process used is dependent upon the function of the software as follows:

- Large complex systems consisting of several programs operating in unison to produce an intended result must be validated.
- For smaller software which only performs numerical manipulation, sample sets of numbers for which results are known should be processed and the results verified. In this case, known results are usually generated by performing hand calculations using the same equations and procedures as the software to verify that the software produces identical results.
- Software which performs as part of instrument operation should be verified as previously described and by processing reference materials through the instrument system. Processed instrument response should be evaluated against expected instrument response and performance.

IT SOPs governing software development and testing include S-ITQ-001, S-ITQ-007, and P-ITQ-013.

6.4.3 Control of Software Changes

STL has a well established process for prioritizing and managing changes to LIMS and LIMS-related software (see S-ITQ-001and S-ITQ-007). Proposals to modify software are written in a Software Enhancement Request, which includes a description of the task to be accomplished, the software to be modified, its functional requirements, and necessary algorithms. The Software Enhancement Request is submitted to the Change Management Committee for approval. The Committee includes representatives from each lab on the QuantIMS network. The Committee establishes a develop schedule and approves the resources needed. Documentation of changes, version control, and historical records of changes is the responsibility of the IT Manager of "Change Management and QA". Because these are modern networked systems, the documentation is kept on the network, rather than keeping redundant records at each facility as GALP suggests. All system software changes are developed in a test area and must pass the designed tests before it is installed in the working area.

The same principles of documenting software changes apply to spreadsheets, small databases, or other small programs that are used solely at the STL Pittsburgh lab. The verification/validation records must explain the functional requirements, the algorithms and formulas used, the testing performed, and are maintained by the lab QA Manager.

6.4.4 Software Maintenance

Software problems are presented to the local LIMS Administrator (LAS) in a Software Problem Report. The LAS presents the issue to a group of the network LASs. The problem is discussed to make sure it is understood, and then a solution is determined and prioritized. Changes to LIMS software for maintenance purposes are announced to each of the QuantIMS locations after revalidating the software.

6.4.5 Software Revalidation

Whenever a program is changed, the change is evaluated to determine if it is significant enough to make revalidation necessary. If features have been added, previous test problems are rerun to demonstrate that their function has not been affected. New test problems are processed, as previously discussed, to verify added performance. If software revision changes the basic operation of the program, complete revalidation of the program may be required.

Spreadsheets and unprotected software used to acquire, process, or report client data must be documented and reverified when changes are made. The test problems used to provide initial verification is reprocessed and the results compared to demonstrate that performance of the software is unchanged.

Laboratory operations is responsible for the generation of the validation and verification documentation for instrument level software. Completed records are provided to QA. STL Information Technology is responsible for generation and maintenance of documentation relating to verification and validation of the STL QuanTIMS system. This is described in SOP P-ITQ-013, Software Quality Assurance.

6.5 Comprehensive System Testing

Comprehensive system testing is performed periodically. Independent auditors, such as Price Waterhouse, include computer systems in their audits, which are commissioned by the laboratory executive management. Extensive testing of all software was performed for the lab's Y2K readiness exercises.

As described in LQM Section 9.4.2, the STL Pittsburgh QA Manager is responsible for ensuring an annual internal audit of all lab areas is performed, including the local IT functions.

6.6 Records Retention

As required by NELAC, electronic raw data and computer documentation are stored for a minimum of five years. See LQM Section 3.0 for further records retention details.

7.0 Contract Review and Project Planning

The generation of environmental analytical data is an intricate process. Success is dependent upon the timely execution of interrelated steps. For many environmental sampling and analysis programs, testing design is site or project specific and is not necessarily the same as the laboratory's standard service. It is STL's intent to provide both standard and customized laboratory services to our clients, provided that any special requirements are documented in writing, and provided performing the work in this manner does not cause the laboratory to violate relevant regulatory requirements. STL Pittsburgh has an organizational system in place to ensure that projects are properly planned prior to project initiation. This means that laboratory personnel understand project requirements, that the client clearly understands the lab's capabilities, that the laboratory has the facilities and resources needed to perform the required tests, that samples will be properly handled, that contingency plans are in place, and that analytical data will be reported in accordance with project needs.

7.1 Contract Review

The process of client request for proposal (RFP) and the laboratory's tender of a written response is a process of communication between both parties to understand project requirements and the laboratory's capabilities. All contracts for new work entered into by STL Pittsburgh are reviewed by the Customer Service Manager (CSM) or designee. Agreements for continuing work are the responsibility of laboratory Project Managers (PMs) or the CSM. Depending on the size and scope of the proposed project, the Laboratory Director and other STL management staff can also be involved. Technical staff (Operations Manager, QA Manager, and IT staff) can be called upon to perform a review of the technical and QA/QC requirements. The CSM or PM, with this internal support, will work with clients to align project requirements with laboratory capabilities. Any contract requirement or contract modification communicated to STL verbally is documented and communicated to the client in writing. Any discrepancy between client requirements and STL's capability to meet those requirements is resolved in writing before acceptance of the contract

All contracts, Quality Assurance Project Plans (QAPP), Sampling and Analysis Plans (SAPs), contract amendments and documented communications become part of the permanent project record as detailed in Section 3.5.

7.2 Certifications and Approvals

A necessary part of the review and work acceptance procedure is the evaluation of project needs for laboratory certification. The persons reviewing the prospective project must determine if project work plans or regulatory permits are tied to specific laboratory certifications or approvals. Where such requirements exist, the laboratory must have the certifications or approvals in place before the work begins. QA personnel coordinate with the state certification agencies to maintain or add additional parameters. Copies of current laboratory certifications are maintained by the QA office, and are available upon request. Table 7.2-1 includes a list of approved parameters for PADEP certification. (Copy of current certificate which includes all methods and analytes is available upon request.)

7.3 Data Collection Process

The sample collection and data generation processes are shown in Figure 7.2-1. These processes are designed to produce analytical data that accurately reflect the nature of the site or sampling point.

7.4 Project Organizational Responsibilities

Each laboratory client is assigned a single point of contact, usually a PM, to ensure that there is a strong line of communication between the client and STL Pittsburgh. As a matter of policy, CSMs or designee, PMs, and Operations Managers work together to accomplish the following prior to receipt of samples at the laboratory:

- Samples are scheduled for arrival at the laboratory
- All unique project requirements have been identified and communicated to all appropriate personnel
- Standardized client, state, federal, or STL programs are appropriately selected
- Fully-qualified subcontract laboratories have been selected if needed
- A review has been performed on all pre-project documents such as proposals, contracts, and/or QAPPs to identify the type of tests required and to ensure project requirements are within the scope of the laboratory being used
- All appropriate and required preparations have been made at the laboratory to accommodate or meet project requirements as described in proposals, contracts, and/or QAPPs
- It has been determined that the laboratory has the capability and the capacity to analyze the samples including equipment, staff, space and workload
- The laboratory is capable of meeting the required sample holding times and is able to report the resulting data within the time line specified by the client
- All known safety hazards associated with the samples have been communicated to all appropriate personnel.

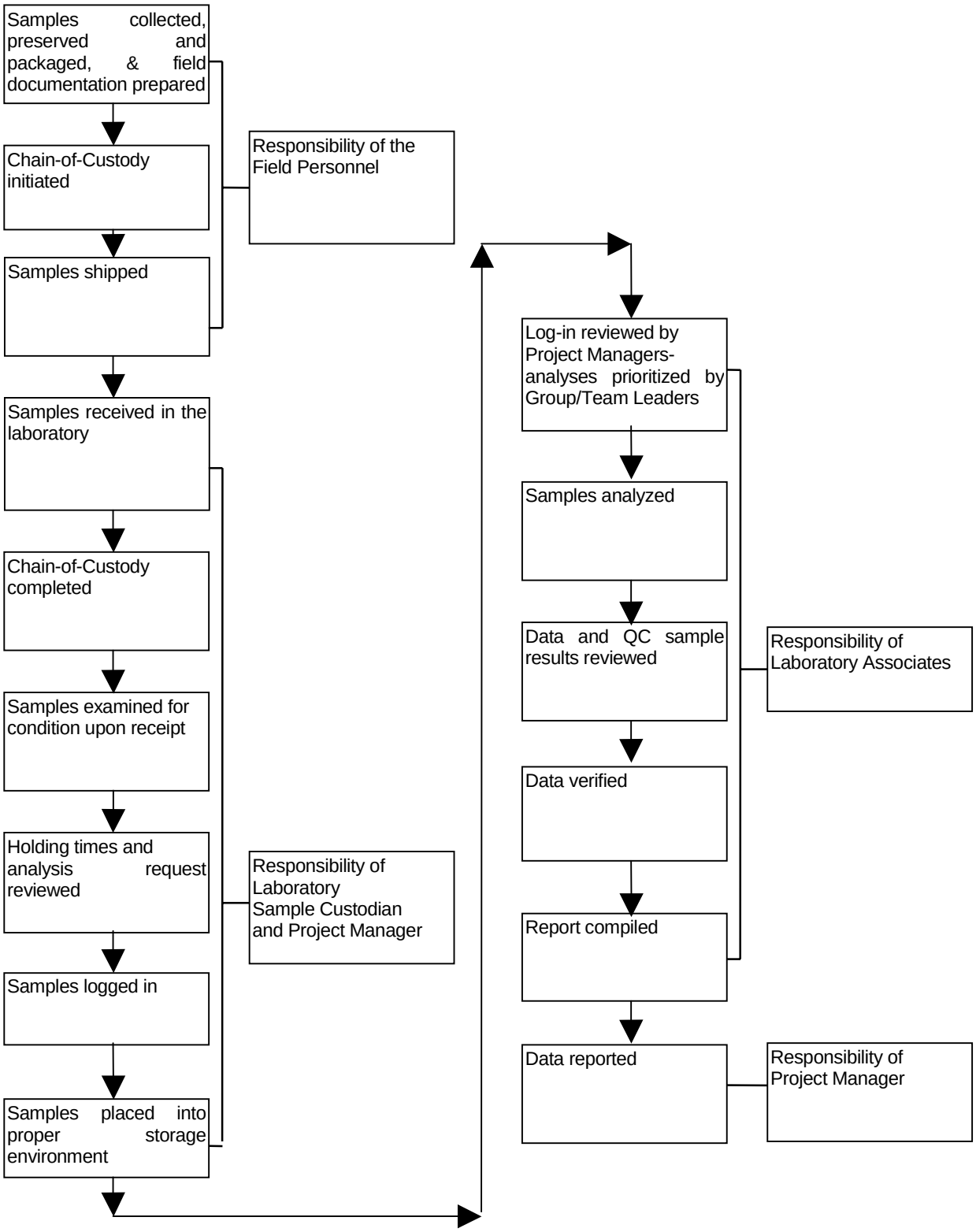
7.5 Communicating Project Requirements Internally

STL Pittsburgh PMs shall document all project-specific requirements prior to receipt of samples. The LIMS system, QuantIMS, requires the PM to enter a “quote” before any samples can be logged in. In addition to price information, the “quote” is a detailed technical specification of the work to be performed. The quote includes identification of project personnel, numbers and types of samples, tests to be performed, reporting limits, QC to be performed, control limits, data qualifier flags to be used, significant figures to be used, and the types of deliverables required. This is the primary means of communicating routine project requirements to laboratory personnel.

Non-routine project requirements are entered into the “Comments” section of the QuanTIMS quote module and are distributed by the sample receiving personnel to each of the operational groups each time samples are logged in and before testing has started. If the special requirements are too lengthy for the quote module comments section, the PM must prepare a Quality Assurance Summary (QAS) or equivalent, which is a written document describing all requirements that are different than routine work. For complex projects, project kickoff meetings are conducted by the PM with each of the operational groups involved.

Figure 7.2-1

Data Collection Process Flow Diagram



7.6 Contingency Planning

An effective QA program must emphasize contingency planning, actions to prevent problems from reoccurring, and to ensure timely and effective completion of a measurement effort. The following are considered relative to contingency planning.

7.6.1 Staffing

A primary objective is to ensure that qualified staff are available to perform the necessary analytical work, regardless of employee turnover, vacation, illness, or other absences. STL Pittsburgh is a relatively large laboratory with multiple staff capabilities for the majority of tests performed. However, other sources of trained personnel are potentially available to assist in the event of unforeseen absences. Given sufficient time for necessary orientation, temporary agency staff can be used. More significantly, STL is a large laboratory network and a large pool of qualified staff can be made available from other STL laboratories.

7.6.2 Backup Instrumentation

Within STL Pittsburgh, duplicate instrumentation is available for most methods to allow uninterrupted work flow if one piece of equipment fails. The laboratory may also choose to lease equipment. However, in circumstances where a catastrophic instrument failure occurs, alternative, but equivalent, methods may be recommended to the client for approval.

- Preventive Maintenance - STL's preventive maintenance program is designed to minimize analytical instrument malfunctions, permit simple adjustments, and to ensure fewer and shorter breakdowns of critical analytical equipment. (See Section 8.11, "Preventive Maintenance and Service".)
- STL Laboratories & Subcontractor Laboratories - To support the laboratory during peak periods or in the event of a critical instrument malfunction, STL has the capability to arrange for the use of other STL laboratories or other qualified analytical laboratories as subcontractors for short-term backup analytical support. **However, use of a subcontractor laboratory must be approved by the client in writing.** For projects requiring NELAC approval, the subcontractor must also be NELAC approved. See Section 5.3 for other procedures related to the control of subcontract laboratory services.
- Uninterruptible Power Supply - An Uninterruptible Power Supply (UPS) system which provides line conditioning and backup power to the LIMS computer system/server. This contingency allows sufficient time for the main computer system to be shut down and for data archival. All electronically generated data that are stored on the main computer system and on individual personal computer (PC) hard drives are backed up at regular intervals. In the event that the main laboratory computer system fails, the analytical data can be retrieved from the PC hard drives.
-

8.0 Work Processes and Operations

Many activities related to environmental projects activities are planned and designed externally to the laboratory or field operation, and are presented to the laboratory in the form of a contract, work plan, sampling and analysis plan (SAP) or QA Project Plan (QAPP). Laboratory and field activities are in turn planned, implemented, and assessed by STL to meet client requirements according to approved procedures and methodologies. The LQM provides the systems to document and implement these activities. The execution and assessment of the implemented operational systems are detailed in STL SOPs. The entire process is assessed on a regular basis for conformance to prescribed requirements.

Standard practices for STL Pittsburgh operations are detailed in this section. Specific project or program requirements that differ from those described here can be met, but they must be explicitly stated in approved contracts, work plans, QAPPs or other project documents. Special project requirements can generally be

accommodated provided that they are properly documented, communicated, and they do not cause the laboratory to violate relevant regulatory requirements.

Table 8.2-3 lists the test methods performed by STL Pittsburgh. Table 8.2-2 lists the SOPs associated with those methods. Table 8.0-1 provides a list of the major equipment in place at the laboratory, and Figure 8.11 (at the end of this Section) shows the laboratory floor plan.

8.1 Traceability of Measurements

STL documents all laboratory activities in sufficient detail to allow their reconstruction. To this end, documentation is generated to trace a sample from its point of origin, through receipt in the laboratory, analysis, reporting and disposal.

The required documentation includes, but is not limited, to:

- Chain of custody documenting movement and possession of samples
- Sample preparation
- Sample analysis
- Calibration and QC data associated with the samples
- Instrument maintenance
- Control of ancillary equipment and materials (e.g., DI water and glassware)
- Sample disposal
- Final reports

These topics are described in this section. Traceability of chemical standards is also discussed in Section 5.2.4.

8.2 Analytical Methods

Whenever possible, STL operations use industry- and regulatory agency-recognized analytical methods from source documents published by agencies such as the Environmental Protection Agency (EPA), Department of Energy (DOE), and the American Society for Testing and Materials (ASTM) as described in STL's SOPs. Analytical methods performed by STL Laboratories are given in Table 8.2-3 lists the methods routinely performed at the laboratory. The methods pending or approved by a NELAC Accrediting Authority are indicated in the table.

Method performance data, as described in Section 8.2.2 below, are developed by the laboratory operations staff to demonstrate method proficiency. The operations staff and the QA staff evaluate and approve the performance data before a methodology is performed routinely. The method must also be described and documented in an SOP.

8.2.1 Standard Operating Procedures

SOPs are required for all repetitive analytical and administrative activities ranging from the receipt of samples in the laboratory through their analysis, reporting, and subsequent disposal. Training, health and safety procedures, QC, method procedures, and instrument and equipment calibrations are included in SOPs. SOP requirements are discussed in the Policy # QA-001, "Standard Operating Procedures". The specifications in the policy meet NELAC requirements. Table 8.2-2 lists laboratory standard operating procedures.

New SOPs and proposed SOP revisions are reviewed by technically qualified lab personnel. SOPs are controlled documents and are distributed and maintained as described in Policy QA-001. Requirements for SOP approval and frequency of review are listed in Tables 2.4-3 and 2.4-4. All significant modifications to the published method are described in a section of the SOP. All operations must be performed as described in these SOPs.

Planned changes in procedure, which may occur due to expected sample matrix effects or project requirements, are documented in the project files. These planned changes may be documented using nonconformance memos, NCMs (see discussion of NCMs in section 9.1), project-specific case narratives, or as modifications or additions to associated QAPPs.

Unplanned deviations in the SOPs, which may occur due to sample matrix or other events, are documented in NCMs and in the project-specific case narratives.

8.2.2 Method Validation and Verification

Before analyzing samples by a new method or method modification, the method must be verified or validated. After which, analyst capability must be demonstrated (see Section 4.4).

8.2.2.1 Method Verification

Method verification is required for methods developed by authoritative agencies, such as EPA or ASTM. The level of verification can vary depending on the type of method or level of modification, but generally should include:

- Determination of method sensitivity,
- Determination of working range,
- An initial demonstration of capability (as specified by NELAC), and
- A written SOP or project-specific written protocol.

Each of these are described in the next section.

8.2.2.2 Method Validation

A complete validation is required for methods developed by STL Pittsburgh. While method validation can take a variety of courses, the following are the key concerns:

- 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. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method
- Determination of Method Sensitivity: Method sensitivity is normally demonstrated using the 40CFR 136B method detection limit protocol (see MDLs, section 8.2.3, below), but can also be based on variance of blank results, and signal-to-noise ratios.
- Determination of Interferences: This is demonstrated by analyzing samples of the matrix of interest that is known to be free of the analyte(s) of interest.
- Determination of Range: In most cases, analytical range is determined and demonstrated by comparison of the response of an analyte at different concentrations to targeted criteria. Often the targeted criteria are represented by the goodness of fit or linearity of the experimental data to a continuous mathematical function or curve. The curve is used to establish the range of quantitation, with the lower and upper values representing the upper and lower quantitation limits. Curves are not limited to linear relationships.
- Determination of Accuracy and Precision: Accuracy and precision studies are generally performed using replicate analysis of samples of known concentration. The resulting percent recovery and relative standard deviation, or other precision measure, is calculated and compared to a set of target criteria.
- Documentation of Method: The method is formally documented in an SOP (see policy QA-001 for details). If a method modification is being performed for a specific short-term project, the modification should be described in a written protocol that is approved by the lab's client, in addition to the in-house approvals required by QA-001.
- Continued Demonstration of Method Performance: Continued ability of the lab to perform the method is addressed in the SOP. Generally this is accomplished with the specified calibration and batch QC requirements.

8.2.3 Method Detection Limits

It is STL Pittsburgh's policy to follow the specification in the U.S. EPA 40 CFR Part 136 Appendix B in determining MDLs for chemical tests. The STL Pittsburgh and DoD requirement for this procedure is further detailed in the STL Pittsburgh SOP PITT-QA-0007 entitled "Determination of Method Detection Limits and Instrument Detection Limits ." This policy requires that the MDLs be determined for each analyte of interest representing the aqueous and solid matrices within the capability of the primary analytical methods.

8.2.4 Instrument Detection Limits

Instrument Detection Limits (IDLs) are required to be performed quarterly for metals constituents and cyanide when analyses are performed in support of Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) activities or when the USEPA CLP SOW protocol is required. IDLs are not required by the SW-846 methods.

When required, IDLs will be performed in accordance with the procedures defined in the applicable USEPA SOW, ILM04.0/ILM04.1/ILM04.1 or subsequent versions, and Policy QA-014, "Determination of Instrument Detection Limits".

Prior to acceptance and use for reporting purposes, all data from detection limit studies and reporting limits must undergo technical review and approval by the laboratory management and QA staff.

8.2.5 Reporting Limits

Reporting limits are established and modified within STL according to the STL Policy QA-009, "Reporting Limits." Two reporting limit conventions are discussed in the policy: the standard Reporting Limit (RL) and the Project-Specific Reporting Limit (PSRL). The standard STL Reporting Limit (RL) is the lowest level at which measurements become quantitatively meaningful. The RL is always greater than the statistically determined MDLs. PSRLs are used when project data quality objectives (DQO) require a reporting limit other than the RL. PSRLs tailor STL's product to meet customer requirements. Higher PSRLs may be established based on maximum contaminant level (MCLs), applicable or relevant and appropriate requirements (ARARs), or project-specific data quality objectives (DQOs). PSRLs below the lab's standard RL may be used, but they must be supported by the MDL and the instrument calibration. A standard at the PSRL taken throughout the entire preparation and extraction procedure may be used to support a PSRL with QA approval. The STL RLs and PSRLs are maintained in the LIMS.

8.3 Data Quality Objectives

Data quality objectives (DQOs) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application (EPA 1994)[1]. Typically, DQOs are identified during project scope and the development of sampling and analysis plans. In this LQM, however, we refer to only the analytical DQOs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory. The EPA has established six primary analytical DQOs for environmental studies. These DQOs are precision, accuracy, representativeness, completeness, comparability, and detectability.

The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. STL incorporates numerous QA and QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA/QC samples and their applications, described in Section 8.4, are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples are received from the client as unknown samples. Analytical laboratory QC samples for inorganic and organic analyses may include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), surrogate spikes, and yield monitors.

8.3.1 Precision and Accuracy

Precision is an estimate of variability, that is, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. The precision of a measurement system is

[1] **"Guidance for the Data Quality Objectives Process", EPA 600/R-96/005, September 1994.**

affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent difference (RPD) for duplicate measurements. Table 8.6-1 illustrates the formulae used to calculate units of precision (i.e., RSD and RPD).

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100).

The precision and accuracy measures that are to be used in evaluating inorganic and organic constituents at STL are provided in Tables 8.4-5 through 8.4-7, in method-specific SOPs, and in the documentation for the analytical method of interest.

Precision and accuracy are determined, in part, by analyzing data from matrix spike and matrix spike duplicates, unspiked duplicates, LCSs, and single blind audit samples. A description of these QC samples is provided in Section 8.4.

8.3.2 Completeness

Completeness is a measure of the percentage of measurements that are judged to be valid measurements. At a minimum, the objective for completeness of data is 90% for each constituent analyzed.

8.3.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result (concentration) is representative of the constituent concentration in the sample matrix. At STL, efforts must be made to analyze an aliquot that is representative of the original sample, and to reasonably homogenize the sample before subsampling.

8.3.4 Comparability

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

8.4 Quality Control Samples

Two types of Quality Control (QC) samples are field QC samples and laboratory QC samples. Field QC samples are collected during the sampling event and are useful in determining sampling precision and accuracy and monitoring for contamination that may occur during collection, transport or storage of environmental samples. Laboratory QC samples are routinely added at the laboratory to the normal sample stream. Successful analysis of these samples demonstrates that the laboratory is operating within prescribed requirements for accuracy and precision. In addition, utilizing matrix-specific laboratory QC samples, information regarding the effect of the matrix or field conditions on the analytical results can be obtained. The following sections describe common field and laboratory QC samples.

8.4.1 Field QC Samples

When field QC sample collection and analysis are required for a project, it is the responsibility of the project sampling supervisor to ensure that this sampling is performed correctly and at the project-required frequencies. Field QC samples may or may not be identified as such to the laboratory and are considered by the laboratory as field samples for the purpose of QC batching, sample preparation and analysis. Field QC sample results are reported in the same manner as actual field samples, unless a specific deliverable is requested by the client. No correction of the analytical data is done in the laboratory based on the analysis of field QC samples.

Field QC sample types, applicability to organic and inorganic analyses, precision and accuracy applications and by whom they are introduced are summarized in Table 8.4-1.

8.4.2 Laboratory QC Samples

Laboratory performance QC is required to ensure the laboratory systems (instrumentation, sample preparation, analysis, data reduction, etc.) are operating within acceptable QC guidelines during data generation as required to meet the client's objectives. Laboratory QC samples consist of method blanks (MB), instrument blanks, laboratory control samples (LCS) and calibration verification samples. In addition to laboratory performance QC, matrix-specific QC is utilized to determine the effect of the sample matrix on the data being generated. Typically, this includes matrix spikes (MS), matrix spike duplicates (MSD), sample duplicates, and the use of surrogate compounds.

Laboratory and matrix-spike QC sample types are summarized in Tables 8.4-2 through 8.4-4. In addition, Tables 8.4-5 through 8.4-7 list laboratory QC samples, acceptance criteria and corrective actions by reference method for inorganic methods, organic methods, and the USEPA CLP Statements of Work respectively. The following sections provide descriptions of laboratory QC samples and their frequency of use. Policy QA-003, "Quality Control Program", describes in detail the QC data evaluation process.

8.4.2.1 Quality Control (QC) Batch

The QC batch consists of a set of up to 20 field samples that behave similarly (i.e., same matrix) and are processed using the same procedures, reagents, and standards within the same time period. This definition of a QC batch is utilized by STL unless there is clear regulatory guidance, contract specifications, or differing client requirements that are explicitly documented. Further details and requirements for the application of the definition of QC batch are described in Policy QA-003.

8.4.2.2 Method Blank

The method blank (MB) is a QC sample that consists of all reagents specific to the method and is carried through every aspect of the procedure, including preparation, cleanup, and analysis. The method blank is used to identify any interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Potential sources of contamination include solvent, reagents, glassware, other sample processing hardware, or the laboratory environment. In general, the method blank is a volume of deionized laboratory water for water samples, or a purified solid matrix for soil/sediment samples, that is processed as a sample. In the event that no appropriate solid matrix exists, deionized water may be used. The volume or weight of the method blank must be approximately equal to the sample volume or sample weight processed. A method blank shall be prepared with each group of samples processed.

8.4.2.3 Instrument/Calibration Blank

The instrument blank is an unprocessed aliquot of reagent used to monitor the contamination of the analytical system at the instrument. System contamination may lead to the reporting of elevated analyte concentrations or false positive data. The instrument blank does not undergo the entire analytical process and generally consists of an aliquot of the same reagent(s) used for a sample dilution. Instrument blanks are also referred to as continuing calibration blanks (CCBs).

8.4.2.4 Laboratory Control Sample

A laboratory control sample (LCS) is a laboratory-prepared suitable clean matrix sample that is fortified with target analytes or a solid reference material purchased from an approved vendor. The LCS contains all target analytes specified in the method, and must contain the same analytes as the matrix spike and matrix spike duplicate. For certain regulatory or client programs, an LCS may contain a full list of analytes. However, in these cases, a subset of analytes, as defined by the program, is used to determine the acceptability of a batch of sample data. The LCS recovery data are used to monitor the analytical method performance in terms of analytical accuracy. On-going evaluation of the LCS recoveries demonstrates that the laboratory is performing the method within statistical control (i.e., accuracy and precision) in the absence of matrix interference. The LCS results, coupled with MS data, help determine whether the laboratory performed the method correctly or the sample matrix affected the analytical results. When a laboratory control sample duplicate (LCSD) is required, a percent recovery for each target analyte is calculated, as well as a relative percent difference (RPD) between the LCS and the LCSD.

8.4.2.5 Matrix Spike

A matrix spike (MS) is an environmental sample to which known concentrations of target analytes have been added. MS samples are analyzed to evaluate the effect of the sample matrix on the analytical methodology. MS samples are generated by taking a separate aliquot of an actual field sample and spiking it with the selected target analyte(s) prior to sample extraction. The MS sample then undergoes the same extraction and analytical procedures as the unfortified client sample. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked and not on samples collected at other locations that are included in the QC batch.

8.4.2.6 Matrix Spike Duplicate

A matrix spike duplicate (MSD) is a second aliquot of a sample that is spiked with the selected target analyte(s) and analyzed with the associated sample and MS sample. The results of the MS and MSD are used together to determine the effect of a matrix on the accuracy and precision of the analytical process. Due to the potential variability of the matrix of each sample, the MS/MSD results may have immediate bearing only on the specific sample spiked and not all samples in the QC batch.

8.4.2.7 Sample Duplicate

A sample duplicate is a second aliquot of an environmental sample taken from the same sample container that is processed identically with the first aliquot of that sample. That is, sample duplicates are processed as independent samples within the same QC batch. The results are compared to determine the sample homogeneity and the precision of the analytical process.

8.4.2.8 Surrogates

Surrogates are organic compounds that are similar in chemical composition and behavior to the target analytes but that are not normally found in environmental samples. Surrogates are added to all appropriate samples and QC samples being tested for organic analytes to monitor the effect of the sample matrix and the procedure on the accuracy of the process.

8.4.2.9 Analytical Spike

An analytical spike is created by spiking target analytes into a prepared portion (i.e., post digestion) of a sample just prior to analysis. It provides information on matrix effects encountered during analysis such as suppression or enhancement of instrument signal levels. It is most often used in elemental analysis involving various forms of atomic emission or atomic absorption spectroscopy. A single analytical spike serves as a single point application of the “method of standard additions” or MSA.

8.4.2.10 Interference Check Sample

An interference check sample (ICS) is a solution containing known concentrations of both interfering and analyte elements. Analysis of this sample can be used to verify background and interelement correction factors.

8.4.2.11 Internal Standards

An internal standard (IS) is a compound or element with similar chemical characteristics and behavior in the analysis process to the target analytes, but is not normally found in environmental samples. The internal standard is usually added after sample preparation. The primary function of the internal standard is quantitation, however, it also provides a short-term indication of instrument performance.

8.5 Data Collection Operations

Laboratory analyses are designed to produce data that are representative of existing conditions present at the time the sample was obtained. The data collection design includes field sampling events, sample handling and custody, analytical operations, data recording procedures, data assessments, data verification, and data reporting requirements and techniques to assess limitations of data use. These operations are discussed in this section through section 8.10.

8.5.1 Field Collection and Shipment

In order to provide a sample that most accurately represents the test matrix, field sample collection personnel must abide by the sample collection guidelines and procedures established by involved regulatory agencies. A significant

part of the efforts of regulatory agencies include the use of "approved" sample containers, chemical and physical preservation techniques, and observance of specified holding times. It is imperative that all samples be collected and preserved according to the appropriate analytical method specified in the QAPP (if one exists). Although the sampling may be performed by non-STL personnel, the importance of sampling and transportation of the sample to the laboratory is understood and must be considered during data validation.

Sampling requirements must be communicated to the sampling team prior to field collection.

Field personnel are responsible for labeling each individual sample collected with the following information:

- Project name
- Unique client sample number
- Sample location (including as appropriate: borehole and depth or grid coordinates)
- Sampling date and time
- Sample preservation
- Analysis required.

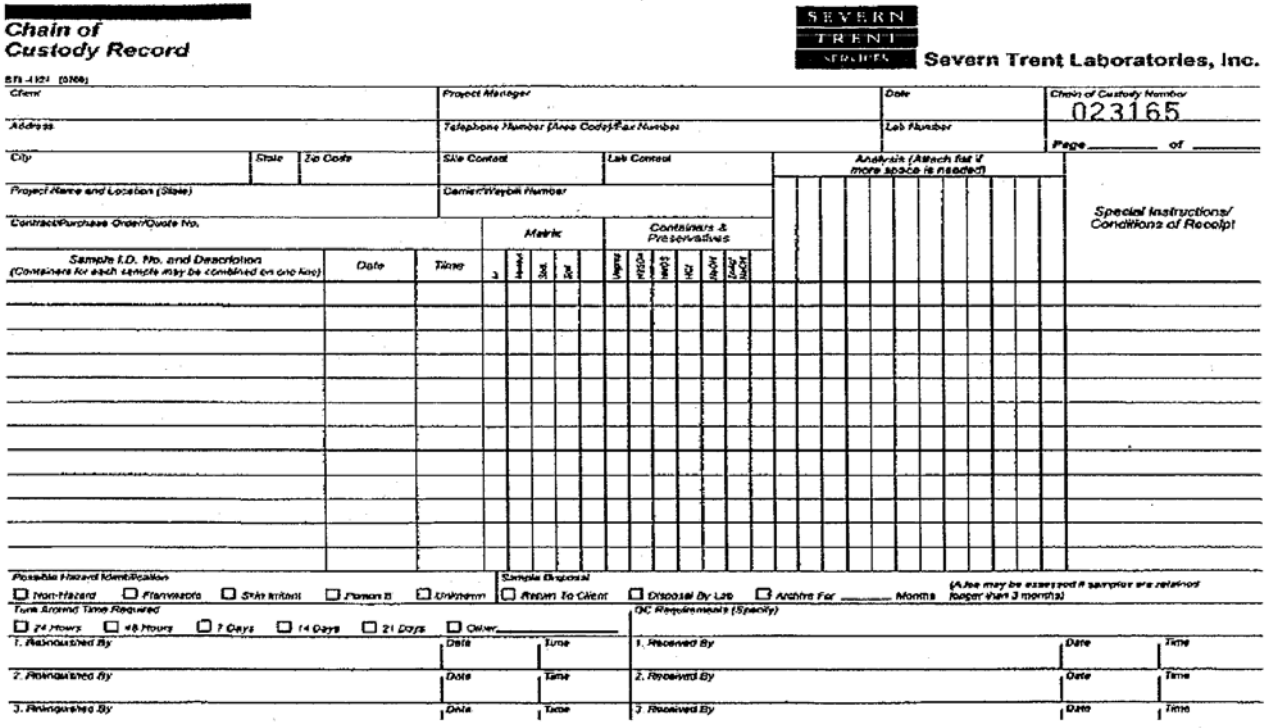
An overriding consideration for the resulting analytical data is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, laboratory custody, and disposal must be documented to accomplish this. Figure 8.5-1 shows an example Chain-of-Custody (COC) form that is used by the STL laboratory to document this evidence. Field personnel are responsible for initiating the COC form.

The prompt shipment of samples to the laboratory is necessary to ensure that required holding times are met. Samples should be shipped by an overnight carrier, be hand-delivered, or transported in a manner that assures prompt delivery to the laboratory. Some sites require an extensive radioactive screening process before a sample may be shipped. In these cases, it is imperative for the Project Manager to maintain good communications with the client to assure proper staffing of the laboratory in response to a decreased holding time.

This is a Controlled Document. When Printed it becomes Uncontrolled.

FIGURE 8.5-1

Example STL Chain-of-Custody Form



Chain of Custody Record

SEVERN TRENT SERVICES — Severn Trent Laboratories, Inc.

Chain of Custody Number: 023165

Page _____ of _____

Client | Project Manager | Date

Address | Telephone Number (Area Code)/Fax Number | Lab Number

City | State | Zip Code | Site Contact | Lab Contact

Project Name and Location (State) | Carrier/Waybill Number

Contract/Purchase Order/Quote No.

Matrix | Containers & Preservatives | Analysis (Attach list if more space is needed) | Special Instructions/ Conditions of Receipt

Sample I.D. No. and Description (Containers for each sample may be combined on one line) | Date | Time

Possible Hazard Identification

☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal

☐ Return To Client ☐ Disposal By Lab ☐ Archive For _____ Months (A fee may be assessed if samples are retained longer than 3 months)

Turn Around Time Required

☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☐ Other_____

QC Requirements (Specify)

1. Relinquished By | Date | Time | 1. Received By | Date | Time

2. Relinquished By | Date | Time | 2. Received By | Date | Time

3. Relinquished By | Date | Time | 3. Received By | Date | Time

8.5.2 Sample Containers, Shipping Containers, Preservatives, and Holding Times

8.5.2.1 Sample Containers

A sample container is defined as the sealed enclosure, usually made of plastic or borosilicate glass that the sample is collected in and stored in until analysis. All sample containers provided by STL operations for environmental sampling are new. All documentation certifying sample container cleanliness must be maintained by the laboratory or the vendor and can be provided to the client upon request. The sample containers to be supplied are listed in Tables 8.5-1 through 8.5-5. Container volumes listed in these tables may be decreased with the approval of the laboratory QA Manager or Technical Director to accommodate reduced sample volumes required by the facility SOP.

8.5.2.2 Shipping Containers

Shipping containers are defined as the sealed enclosure in which the sample containers are stored during shipment from the sample collection site to the analytical laboratory. Shipping containers must be of sufficient number and size to accommodate the samples in an upright condition. Shipping containers must also meet all requirements for the shipment of environmental and/or radioactive samples.

Packaged samples must be shipped to the analytical laboratory in a safe manner that preserves the integrity of the samples. The most common method of sample shipment employs coolers or ice chests that are sealed with custody tape and shipping tape. These coolers must be durable and resistant to crushing during shipment. All coolers must be well maintained and cleaned to prevent cross-contamination of the samples. It is the ultimate responsibility of the person collecting and packaging the sample for shipment to ensure that the shipping containers are clean and functional.

To help prevent sample breakage during shipment, additional consideration must be given to providing shock absorbency to all samples packaged inside the shipping container. Use of bubble-wrap around each sample container is the best way to provide this protection. Foam packing materials and vermiculite are also successfully used.

8.5.2.3 Sample Preservatives

Most analytes have a finite holding time in a given sample matrix. Sample preservation is the chemical or physical means by which samples are treated during and/or following sample collection to aid in the stability of the analytes of interest in that matrix. Sample holding times are also adversely affected when samples are improperly preserved, or shipped unpreserved. The preservation of samples at the time of sample collection will follow the requirements of the analytical methods used. This preservation includes the addition of reagents to deter chemical and biochemical degradation and the maintenance of refrigeration during transit and ultimate storage in the laboratory. The required preservatives for the analysis to be performed on each matrix are included in Tables 8.5-1 through 8.5-5.

8.5.2.4 Sample Holding Times

Holding time is defined as the maximum allowable time a sample can be stored after sample collection and preservation (or laboratory receipt for CLP) until appropriate processing occurs (preparation or analysis). The holding time may vary according to method or client requirements. Tests designated with holding times as “analyze immediately or ASAP” are considered parameters that should be tested by field personnel or on-site. Each operation has a system in place to ensure that holding times are monitored by each group within the operating unit. It is the responsibility of each STL associate processing the sample to assure that holding times are met. STL is responsible for meeting all holding times for properly preserved samples received within 48 hours of collection or if less than half the holding time has passed. If these conditions are not met, STL will attempt to expedite sample analysis as soon as possible.

Sample holding times are listed in Tables 8.5-1 through 8.5-5.

8.5.3 Sample Handling

STL Pittsburgh’s SOP PITT-QA-0051 describes the sample receipt and log-in process in detail. The following sections describe the general policies followed by STL.

8.5.3.1 Sample Receipt

Samples shall be received and logged in at STL by a designated sample custodian or other properly trained associate. Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment. At a minimum, this consists of gloves, a lab coat, and safety glasses
- Examine the shipping containers to verify that the custody tape is intact
- Examine all sample containers for damage
- Open shipping containers in adequately ventilated areas to assure worker safety
- Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the COC
- Compare samples received against those listed on the COC
- Verify that sample holding times have not been exceeded
- Examine all shipping records for accuracy and completeness
- Determine sample pH (if required for the scheduled analysis) (except VOA samples) and record on the COC
- Sign and date the COC immediately (only after shipment is accepted) and attach the waybill
- Note any problems associated with the coolers and samples on the COC, immediately initiate a Condition Upon Receipt Report (CUR) or equivalent format, and notify the PM who in turn notifies the client
- Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test
- Place the samples in proper laboratory storage.

A CUR or an equivalent form/system is generated by sample control during the sample log-in process to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The affected client shall be notified by the PM or designee of all CURs generated for their samples. The PM is responsible for resolving with the client how to proceed with the samples and documenting the decision to proceed with the analysis of compromised samples. CURs must be resolved prior to sample preparation and analysis. The completed CUR form shall be stored in the project file. An example CUR is shown in Figure 8.5-2. The report narrative will include an explanation of sample receiving related anomalies. Further details are given in SOP No. PITT-QA-0051.

8.5.3.2 Exceptions or Discrepancies

STL reserves the right to reject samples for any of the following reasons:

- No custody seals as required by project
- No chain of custody documentation provided
- Preservation inappropriate for analysis requested
- Sample container inappropriate for analysis requested
- Sample received out of holding time for analysis requested
- Incomplete sample information provided
- Discrepancies between COC and sample labels
- Samples have high levels of polychlorinated dibenzo-p-dioxins/ dibenzo furans (PCDD/PCDFs)

- Samples have a high level gross alpha or beta radiation
- Samples are from a site known to contain chemical warfare agents (CWAs) and the samples have not been screened for them.
- Samples are Asbestos containing material.

These or any other project exceptions or discrepancies are discussed with the client and agreed upon action taken.

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FIGURE 8.5-2

Example STL Condition Upon Receipt Anomaly Report (CUR)

Condition Upon Receipt Variance Report
STL Pittsburgh Laboratory

Client: _____ Date: _____
Project No.: _____ Initiated by: _____
Analysis Requested: _____ RFA/COC: _____
Client Sample Numbers Affected: _____

Condition/Variance (Check all that apply):

1. ☐ Not enough sample received for proper analysis. Received approx. _____
2. ☐ Sample received broken/leaking.
3. ☐ Sample received without proper preservative.
   ☐ Cooler temperature not within 4°C ± 2°C.
   Record temperature: _____
   ☐ pH _____
   ☐ other: _____
4. ☐ Sample received in improper container.
5. ☐ Sample received without proper paperwork.
   _____
6. ☐ Paperwork received without sample.
7. ☐ No sample ID on sample container.
8. ☐ Custody tape disturbed/broken/missing.
9. ☐ Sample splits performed by lab.
10. ☐ Volatile sample received with approximately _____ mm headspace.
11. ☐ Sample ID on container does not match on paperwork. Explain:
    _____
    _____
12. ☐ All coolers on airbill not received with
13. ☐ Other (explain below):
    _____
    _____
    _____

Notes: _____
_____
_____

Corrective Action:

☐ Client's Name: _____ Informed verbally on: _____ By: _____

☐ Client's Name: _____ Informed in writing on: _____ By: _____

☐ Sample(s) processed "as is" _____

☐ Sample(s) on hold until: _____ If released: _____

Sample Control Supervisor Review: _____ Date: _____

Project Management Review: _____ Date: _____

SIGNED ORIGINAL MUST BE RETAINED IN THE PROJECT FILE

STL PT/06-02/96-005/CURVR.DOC

8.5.3.3 Sample Log-In

Sample log-in activities at STL are fully documented in SOP PITT-QA-0051. The following is a general description of the log-in process:

- Enter the samples in the laboratory sample log-in book, and/or the LIMS which contains the following information at a minimum:
- Project name or identification number
- Unique sample numbers (both client and internal laboratory)
- Type of samples
- Required tests
- Date and time of laboratory receipt of samples
- Field ID supplied by field personnel
- Notify the PM and appropriate Group/Team Leader(s) of sample arrival
- Place the completed COCs, waybills, and any additional documentation in the project file.

8.5.3.4 Sample Storage

The primary considerations for sample storage are:

- Maintenance at the method prescribed temperature, if required
- Maintenance of sample integrity through adequate protection from contamination from outside sources or from cross-contamination of samples. Low-level and high-level samples, when known, must be stored separately. Samples and standards must be stored in separate refrigerators or freezers. Storage areas for volatile organic test requests should be monitored twice per month by the analysis of a holding (refrigerator) blank (an aliquot of contaminant-free water stored in a VOA vial)
- Security of samples within the laboratory.

The requirements listed in Tables 8.5-1 through 8.5-5 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of sample control personnel. STL will assign individuals the responsibility of notifying the Group/Team Leaders or their designees if there are any samples which must be analyzed immediately because of holding time requirements.

8.5.3.5 Internal Sample Chain-of-Custody and Interlaboratory Transfers

Sample custody within STL laboratories is described in SOP PITT-QA-0051. Internal COC may be required for programs defined by state or federal agency. The sample custody documentation shall include the following minimum requirements:

- Name of associate taking custody of the sample from the sample storage area for preparation or analysis
- Dates sample removed from and returned to the sample storage area
- Identification of tests to be performed on the sample aliquot(s) selected by the associate
- Sample matrix
- Laboratory sample numbers
- Sample storage location.

Additional custody records can be provided by the laboratory; at the specific request of the client. Access to STL is restricted to prevent any unauthorized contact with samples, extracts, or documentation.

Samples transferred to a different laboratory than the original receiving facility are transferred under chain-of-custody (COC). The COC is maintained whether the laboratory is another STL facility or a subcontracted laboratory. If the entire sample volume is transmitted, the original copy of the client's COC form will be used to document the relinquishing of the sample and will accompany the sample to its destination. A copy of the completed COC form shall be retained in the laboratory project file. In the case where an aliquot of a sample is shipped from the laboratory, a new COC will be generated by the laboratory and shipped with the sample aliquot. The original COC will be retained in the project file at the site holding the original sample container.

Samples are not transferred to other STL facilities or to subcontractor laboratories without prior approval of the client.

8.5.3.6 Subsampling

Sample preparation procedures are referenced in the method SOPs. Sample subsampling will be performed in accordance with the associated sample prep SOPs and SOP PITT-.QA-0024, Subsampling.

8.5.3.7 Sample Disposal and Return Chain-of-Custody

After the requested analyses on the samples have been completed, any remaining portions of the samples will be maintained by the sample custodian until the samples are disposed of or returned to the client. The disposal of each sample is recorded on the client's COC form, in LIMS, or referenced in the project file. Sample disposal procedures and documentation are described in operation-specific SOPs. STL's routine sample retention period is at least thirty days after the analytical report is issued to the client, unless otherwise specified by the client.

If samples are returned to the client rather than disposed of by the laboratory, the original COC or a new COC is used to document custody transfer back to the client from the laboratory. A copy of the completed COC is retained in the laboratory project file.

8.5.4 Calibration Procedures and Criteria

All equipment and instruments used at STL operations for quantitative measurements are controlled by a formal calibration program. Table 8.0-1 lists the lab's major analytical instrumentation , and Tables 8.5-6 through 8.5-8 outline calibration requirements. Calibrations may be periodic or operational. These are described in the lab's method SOPs. STL Corporate Policy P-T-001, "Selection of Data Points" is applicable when the number of data points is not described in the method. At a minimum, these calibration procedures shall include:

- Instrument to be calibrated
- Reference standards used for calibration
- Calibration technique (e.g., linear, quadratic)
- Acceptable performance tolerances and corrective actions required if specifications are not met
- Frequency of calibration
- Calibration documentation requirements.

Whenever possible, recognized procedures such as those published by ASTM or the USEPA or procedures provided by manufacturers shall be adopted. If established procedures are not available, a procedure shall be developed considering the type of equipment, stability characteristics of the equipment, required accuracy, and the effect of operation error on the quantities measured.

8.5.4.1 Physical Reference Standards

Physical reference standards associated with periodic calibrations include weights for calibrating balances and certified thermometers for calibrating working thermometers. Whenever possible, physical reference standards shall be calibrated by a body that can provide traceability to nationally or internationally recognized standards. If these standards are not available, the basis for the reference standard shall be documented.

Physical reference standards shall be used only for calibration procedures and shall be stored separately from equipment used for analysis.

8.5.4.2 Chemical Reference Standards and Reagents

Chemical reference standards are generally associated with operational calibration. These standards include reference materials traceable to recognized standards suppliers. This may include vendor-certified materials traceable to national or international standard reference materials (e.g., NIST). This topic is also discussed in the Section on "Procurement of Supplies and Services" (see 5.2.4).

All chemical reference standards maintained in the laboratory for use in calibrations (or as QC spiking solutions) and reagents prepared in the laboratory shall be labeled or referenced to appropriate documentation (hard copy or electronic) with the following information at a minimum:

- A unique identification including concentration (solutions containing several analytes can be identified such that the solution constituents and concentrations can be referenced to a logbook)
- Medium prepared in
- Preparation date
- Expiration date
- Initials of preparer.

Vials containing standard solutions that are not large enough to accommodate labels listing the above information may be referenced to a laboratory logbook/ notebook entry or standards software. The expiration date of the working standard and reagent must not exceed the expiration date of the original material. These records should provide sufficient detail to allow one to reproduce the standard or reagent.

Records for all purchased standards and reagents shall include the date of receipt, the date opened, and, where applicable, the expiration date.

8.5.4.3 Standard Verification

When possible, reference standards are purchased from a STL preapproved vendor. Standards are verified by quantitation against a second known standard before reporting data. The standard for verification must meet the laboratory's criteria for the independent/second source ICV verification. Therefore, the verification of a new standard initial calibration with a second source ICV meets this verification requirement. Realizing that some "bad acting" analytes may not meet these criteria and must be approved by the QAM before use. Standard spiking solutions and surrogates shall be verified by analyzing an LCS with the new standards and verifying against historical criteria limits. Special standards that are obtained from another source must also be independently verified at the lab. Verification by the laboratory of a reference standard from neat materials is also necessary.

To extend the use of an expired standard, which may not be allowed by all programs, reverification is necessary provided that new analysis produces acceptable data. The verification of an expired standard is performed against a current, independent standard reference material by analyzing within a valid calibration and QC.

Stock and working standards and reagents are checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. Care is exercised in the proper storage and handling of standard and reagent solutions. Standards and reagents are always stored separately from samples.

An independent or second source standard is used to verify initial calibrations. An independent/second source standard is defined as a standard composed of the same target constituents as, but from a different source than those used in the standards for the initial calibration. An independent standard may be a laboratory-prepared or a certified independent standard solution(s). Independence of reference material can be achieved by: (1) purchasing reference materials from two separate vendors, (2) using a different lot from the same vendor that is certified by the vendor as an independent standard or (3) having two separate individuals prepare the calibration and verification standard solutions if independent sources are not available.

8.5.4.4 Periodic Calibration

Periodic calibration is performed at prescribed intervals. In general, equipment that can be calibrated periodically is a distinct, singular purpose unit and is relatively stable in performance. These include balances, micropipettors,

counters, thermometers, refrigerators, freezers, and ovens. Equipment employed at STL requiring periodic calibration are listed along with their respective calibration requirements in Tables 8.5-6 through 8.5-8. NELAC requires mechanical volumetric dispensing devices (except Class A glassware) to be checked for accuracy or at least a quarterly basis if in use. The laboratory unit has an SOP in place for the calibration of this equipment if in use at their location.

8.5.4.5 Operational and Continuing Calibration

Operational calibration is routinely performed as part of instrument usage, such as the development of a standard calibration curve (see Tables 8.5-6 to 8.5-8). The accuracy of initial calibrations are to be verified prior to sample analysis through the use of an independent standard in situations where the source method requires calibration verification.

Detailed requirements for operational and continuing calibration are contained in method-specific SOPs.

When an initial calibration is not performed on the day of analysis, the validity of the initial calibration verification must be verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch.

- A continuing instrument calibration verification must be repeated at the beginning and end of each analytical batch. However, if an internal standard is used, the continuing calibration verification need only be run at the beginning of each analytical "run period" (for example: a 12 hour tune clock for SW-846 GCMS). For methods which do not employ internal standards, a calibration check standard will be run at the start of each run sequence and for NELAC compliance this check standard will be at a concentration differing from the continuing calibration verification standard used throughout the remainder of the sequence.
- Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, and calibration curve or response factor.
- If the continuing instrument calibration verification results obtained are outside the established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications or a new initial instrument calibration must be performed. If the laboratory has not demonstrated acceptance performance, sample analyses must not occur until a new initial calibration curve is established and verified. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:
- When the acceptance criteria for the continuing calibration verification is 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 continuing calibration verification is exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit. Otherwise the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

8.5.4.6 Calibration Failure

Equipment or instruments that fail calibration or become inoperable during use shall be tagged to indicate they are out of calibration. Such instruments or equipment shall be repaired and successfully recalibrated before reuse. Following recalibration or verification, back to control will be documented in the injection/run log and/or maintenance logbook through the routine identification of the required calibration runs specified by the standard operating procedure.

8.5.4.7 Calibration Records

Calibration shall be documented for each piece of equipment subject to calibration. All calibration records (periodic and operational) directly affect data and may not be limited to one project. These records shall be stored in either the quality records or the associated project files. Project files that include sample data shall either include the calibration records or include reference to them.

8.6 Quality Assessment

The effectiveness of the QA practices is measured by the quality of data generated by the laboratory. Procedures are in place to detect, prevent, and correct quality problems and to ensure quality improvement. Items and processes that do not meet established requirements must be investigated to determine their cause. Improvements must be implemented in the operations that will prevent a recurrence of these quality problems and provide overall quality performance. All phases of laboratory work should be designed with the objective of preventing problems and improving quality on a continuous basis.

8.6.1 Data Quality Assessment

Data quality is judged in terms of precision, accuracy, representativeness, completeness and comparability. The areas of representativeness, comparability, and completeness for an overall project, inclusive of sampling issues, may be beyond the control of the laboratory. The elements over which the laboratory has direct control are precision, accuracy, and completeness relative to analytical testing results.

Precision and accuracy assessments are made as part of the evaluation of laboratory QC data generated during sample preparation and analysis. The QC samples employed at STL Pittsburgh as part of routine sample analysis are summarized in Section 8.4 of this document. Table 8.6-1 shows the precision and accuracy measurements employed. Analytical method SOPs and STL Policy Number QA-003 include information on requirements for the type of QC samples, frequencies, and acceptance criteria. Additionally, the SOPs and Policy describe the appropriate actions to be taken when a QC sample result does not meet acceptance criteria. Statistical Evaluation of Data In-house limits for all QC data must be evaluated at least annually and compared to the limits published in the methods for applicable matrices. Method limits will be employed until sufficient QC data are acquired. A minimum of 20 to 30 data points are recommended to establish the in-house QC limits. If in the judgment of the QA Manager the method limits are sufficiently strict, they may be used in lieu of the in house calculated limits. However, this will be done on a test basis rather than a compound specific basis. Calculated results of the QC (LCS) samples are evaluated by comparing against control limits (3-sigma).

Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the non-conformance system, to monitor for trends. Program-specific data analysis requirements for control charts are followed as required for data generated under those programs. These additional requirements shall be documented in a QAPP or QAS.

Precision and accuracy measurements employed by STL Pittsburgh are shown in Table 8.4-3 through 8.4-7. Calculated results of these QC samples are evaluated using statistical tables or control charts.

8.7 Data Recording Procedures

To ensure data integrity, all documentation of data and records generated or used during the process of data generation must be performed in compliance with SOP Number QA-008, “Data Recording Requirements”.

8.8 Data Reduction and Verification Procedures

Data review procedures comprise a set of computerized and manual checks applied at appropriate levels of the measurement process. Data review begins with the reduction or processing of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to the final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst who performs a 100 percent review of the data to ensure the work was done correctly the first time. Data verification continues with review by a second reviewer who verifies that data reduction has been correctly performed and that the analytical results correspond to the data acquired and processed. This procedure is outlined in Figure 8.8-1.

8.8.1 Data Reduction and Initial Verification

Data reduction and initial verification may be performed by more than one analyst depending upon the analytical method employed. The preparation and analytical data may be reviewed independently by different analysts. In these instances, each item may not be applicable to the subset of the data verified or an item may be applicable in both instances. It is the responsibility of the analyst to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:

- Sample preparation information is correct and complete including documentation of standard identification, solvent lot numbers, sample amounts, etc.
- Analysis information is correct and complete including proper identification of analysis output (charts, chromatograms, mass spectra, etc.)
- Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers
- The appropriate SOPs have been followed and are identified in the project records
- Proper documentation procedures have been followed
- All nonconformances have been documented
- Special sample preparation and analytical requirements have been met.
- The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or otherwise specified by the client.

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.

Manual integrations are sometimes necessary to appropriately evaluate chromatographic data, but must only be performed when necessary. Further discussion of manual integrations and the required documentation is given in Policy Number S-Q-004, “Acceptable Manual Integration Practices”.

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.

If data are directly acquired from instrumentation and processed, the analyst must verify that the following are correct:

- Project and sample numbers
- Calibration constants and RFs
- Units
- Numerical values used for reporting limits.

Analysis-specific calculations for methods are provided in SOPs. In cases where computers perform the calculations, software must be validated or verified, as described in Section 6.0 of this document, before it is used to process data.

The data reduction is documented, signed and dated by the analyst completing the process. Initial verification of the data reduction by the same analyst is documented on a data review checklist, signed and dated by the analyst. Data review requirements are described in Section 5.3.6 of the QMP.

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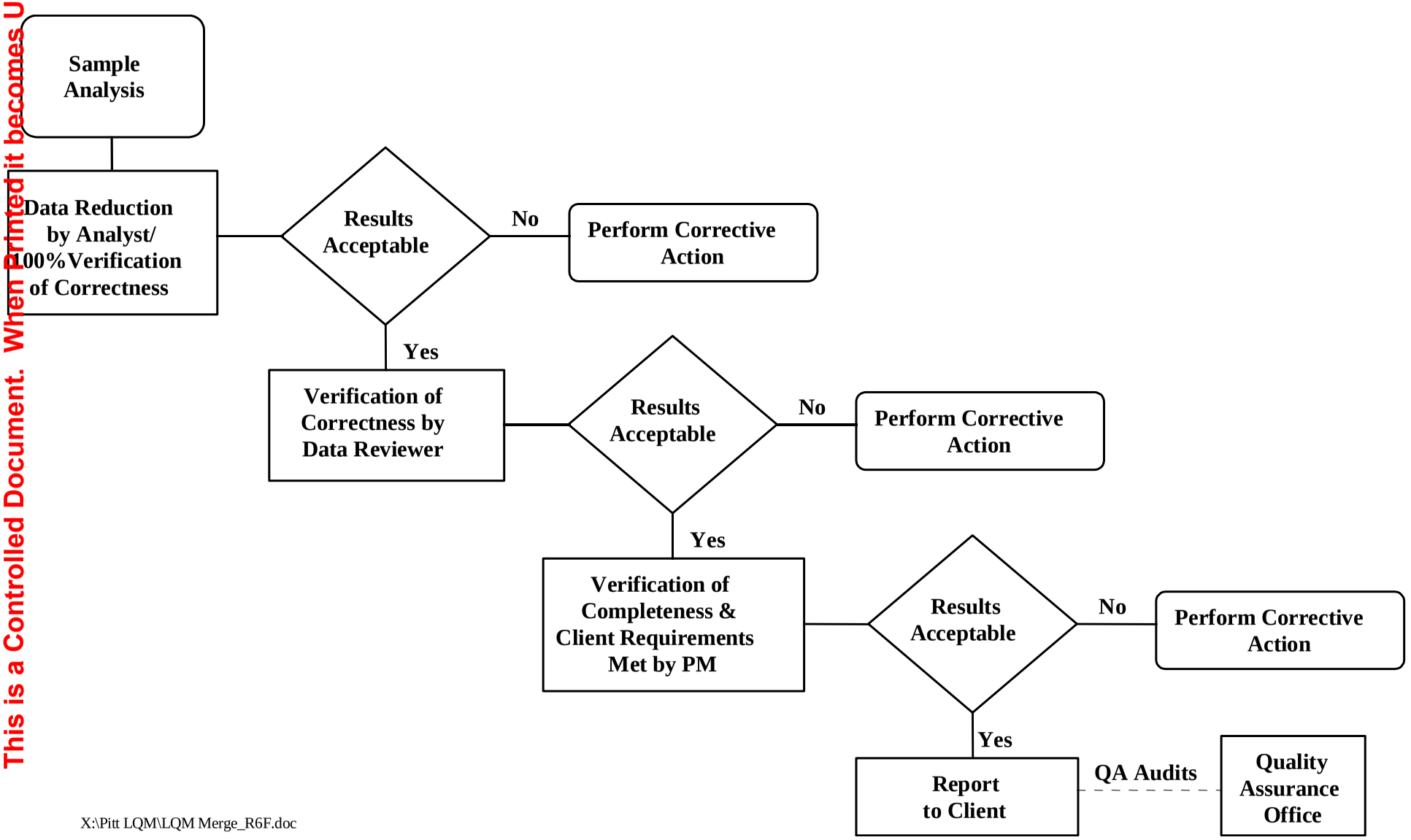
FIGURE 8.8-1

Data Reduction, Verification, and Reporting



8.8.2 Data Verification

Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through Level 1 review is performed by an experienced peer, supervisor, or designee. This check is performed to ensure that level 1 review has been completed correctly and thoroughly. The second level reviewer examines the data signed by the analyst. This review includes an evaluation of all items required in the raw data package. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:

- Adherence of the procedure used to the requested analytical method SOP
- Correct interpretation of chromatograms, mass spectra, etc.
- Correctness of numerical input when computer programs are used (checked randomly)
- Correct identification and quantitation of constituents with appropriate qualifiers
- Numerical correctness of calculations and formulas (checked randomly)
- Acceptability of QC data
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.)
- Documentation of dilution factors, standard concentrations, etc.
- Sample holding time assessment.

This review also serves as verification that the process the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and specific instructions given on the project QAS or equivalent summary form
- Nonconforming events have been addressed by corrective action as defined on a nonconformance memo
- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct
- The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.

The specific items covered in the second stage of data verification may vary according to the analytical method, but this review of the data must be documented by signing the same checklist. Data review requirements are described in Section 5.3.6 of the QMP.

8.8.3 Completeness Verification

A third-level review is performed by the PM. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG)
- Every parameter or target compound requested is reported with either a value or reporting limit
- The correct units and correct number of significant figures are utilized
- All nonconformances, including holding time violations, and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory

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- The final report is legible, contains all the supporting documentation required by the project, and is in either the standard STL format or in the client-required format.
- Implement checks to monitor the quality of laboratory results using correlation of results for different parameters of a sample (for example, does the TOC results justify the concentration of organic compounds found by GC/MS.)
- A narrative to accompany the final report will be finalized by the PM. This narrative will include relevant comments collected during the earlier reviews.

8.9 Data Reporting

8.9.1 Data Reports

STL Pittsburgh is capable of developing a variety of data deliverable reports. Standard reports will contain:

- Cover Letter/Narrative - Information on sample types, tests performed, any problems encountered, and general comments are provided.
- Analytical Data - Data are reported by sample or by test with the appropriate significant figures and reporting limits, and have been adjusted for dilution, if appropriate. Pertinent information including dates sampled, received, prepared, extracted, and analyzed are provided.
- Laboratory Performance QC Information - The results of LCSs and method blanks analyzed with the project are listed. Any data or QC anomalies are discussed in the narrative.
- Matrix-Specific QC Information - Results of any sample duplicates and MS/MSDs analyzed with the samples as batch QC are reported. Other project-specific QC requested by the client are also reported. The results include supporting information such as amount spiked, percent recovery, or percent difference/RPD.
- Methodology - Reference for analytical methodology used is cited.
- Other Deliverables - Other deliverables available include disk deliverables, electronic data transfer, sample raw data packages, complete deliverable packages, and custom report formats. Requirements for electronic reporting are defined in Policy QA-017, “Electronic Reporting”.

8.9.2 Final Report Details

STL Pittsburgh will provide paginated reports or a uniquely defined, identifiable certificate/report (i.e. electronic file, CD). The report will include:

- Report title, name, address and phone number of the laboratory.
- Name and address of client/project name/client identification number.
- Description (lab ID of sample).
- Dates and Time of sample collections (if known), receipt, preparation and analysis.
- If the required holding time is 48 or less, time of sample preparation and analysis.
- Method identifiers traceable to all procedures used.
- Reporting limit.
- Test result with appropriate units and how reported (wet weight/dry weight). Also identify any results outside of quantitation limits. When required, a statement of the estimated uncertainty of the test result should be added.
- If appropriate, description of any QC failures or deviations from SOPs.

- Signature and title of the individual responsible for the report. Electronic signature is acceptable.
- Date of issue.
- All subcontract work must be clearly identified, and name and address of outside subcontractor noted.
- here relevant, a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory
- Where relevant, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory.

After final report any correction, addition, or deletion must clearly identify its purpose and meet the above reporting requirements as appropriate.

All applicable elements from above should be available for review if not issued in a formal report by an in-house or captive laboratory.

8.9.3 Verbal Results

STL Pittsburgh, as a policy, discourages the release of data verbally or without full data review. If however, the client requests analytical results to be communicated verbally or by facsimile prior to final review, they must be clearly identified as “Preliminary” results. The client must understand that the data have not undergone the required levels of review and may potentially change.

8.9.4 Reporting Analytical Results

Sample results are reported according to analytical method SOPs or client specifications. Normally, the laboratory uses the STL Pittsburgh Reporting Limit (RL) at which any analyte of interest detected at or above that level is reported as a positive value and any analyte of interest not detectable or detected below that level is reported as “not detected” at the RL. The laboratory will normally report results within the calibration, however, any reported results outside of the calibration range will be documented in the final report.

If a QC measurement is out of control and the data is to be reported, data qualifiers are reported with samples associated with failed QC measurements.

The laboratory must certify that the test results meet all NELAC requirements or provide reasons and/or justification if they do not.

In some cases a contract, QAPP, or documented client request may require the laboratory to report sample results in a specified manner. Some examples are given below:

- The laboratory may be requested to report all analytes of interest that are less than the laboratory's RL but are greater than the MDL. This data will be flagged with an appropriate qualifier or noted in the report case narrative. (See precautions in “Establishing Reporting Limits”, Policy Number QA-009).
- The laboratory may be requested to report any tentatively identified compounds (TICs). These data will be flagged with an appropriate qualifier.
- The laboratory may be requested to report sample results using an RL that is higher than their normal level. In this case, only the analytes of interest found at or above that level would be reported as positive values. In this case, the laboratory will state the PSRL rather than the RL. All analytes of interest not detected or detectable below that level would be reported as “not detected” at the PSRL.

In this situation, the laboratory must include documentation in the project file that supports the reporting procedure employed.

It is the responsibility of the laboratory to provide for a reporting system that assures that any problems associated with an analysis are properly documented on a nonconformance memo, communicated to the appropriate STL Pittsburgh staff, and addressed appropriately in the data report.

8.9.5 Reissued Deliverables

If, after issuance of a report, STL Pittsburgh observes any mistake that affects the results reported or the QC interpretation of those results, the client will be notified. After issuance of the report, the laboratory report remains unchanged. Any material amendments to a report after issue made only in the form of a further document, or data transfer must include the statement “Supplement to Test Report” or “Revised” or otherwise identified. Where a report is revised both the original and the revised report will be archived and clearly identified.

8.9.6 Client Confidentiality

Data and sample materials provided by the client or at the client’s request, and the results obtained by STL, shall be held in confidence, unless such information is generally available to the public or is in the public domain. STL’s reports, and the data and information provided therein, are for the exclusive use and benefit of our clients, and are not released to a third party without written consent from the client. Data confidentiality is also discussed Section 3.6.

8.10 Data Validation

Data validation for STL refers to data reviews conducted in accordance with the

USEPA CLP "Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses" and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses", or modifications thereof, for non-CLP type analyses.

This form of data validation provides an impartial evaluation of the laboratory's results. Data validation may be requested by the client for a percentage of data and is usually performed by a third party, one which was not involved with the sample analysis. Qualifiers are assigned to data, when required, according to the requirements of the data validation protocol being used.

8.11 Preventive Maintenance and Service

Facilities, instruments, equipment, and parts are subject to wear, deterioration, or change in operational characteristics. Within STL, preventive maintenance, coupled with vendor service agreements, is an organized program of actions taken to maintain facilities and equipment in control.

8.11.1 Analytical Instrumentation and Equipment

The primary purpose of the maintenance program is to prevent instrument and equipment failure and to minimize down time. A properly implemented maintenance program increases the reliability of a measurement system.

Each instrument or piece of equipment shall be uniquely identified. The laboratory maintains the following:

- Instrument/equipment inventory list
- Instrument/equipment major spare parts list or inventory
- External service agreement documents (if applicable)
- Instrument-specific preventive maintenance logbook or file for each functional unit.

The records of routine maintenance and non-routine maintenance shall include at a minimum:

- Name and serial number of the item or equipment
- Details of maintenance performed
- Dates and results of recalibrations/ reverifications indicating return to control
- Analyst initials and the date maintenance was performed whether by the analyst or a contracted service representative.

Any item or equipment that does not perform to specifications or defective shall be taken out of service, and tagged as out of service until it has been repaired and shown by calibration/ verification to perform satisfactorily.

8.11.2 Frequency of Equipment Maintenance

The frequency of maintenance must consider manufacturer's recommendations and previous experience. Frequency of preventive maintenance along with the recommended preventive maintenance schedules are given in Tables 8.11-1 through 8.11-30 for analytical instrumentation and equipment or defined in operation specific routine maintenance SOPs. Frequency of maintenance for the facility systems is documented in the CHP.

8.11.3 Facilities

Another important aspect of the laboratory operation is the existence and maintenance of adequate, safe, and clean facilities including appropriate engineering controls such as proper ventilation, lighting, dust control, hoods, air flow, protection from extreme temperatures, waste disposal, and a source of stable power. The facility floor plan is provided in Figure 8.11.

The maintenance and use of these facilities and proper operations are described in the Chemical Hygiene Plan (CHP). The Laboratory Director has responsibility for ensuring a properly maintained facility. The Laboratory Director also has the responsibility for ensuring that facilities are available to store samples properly without contamination, work areas are equipped with adequate bench, hood and operational space, and that procedures are in place to ensure the areas are free from chemical contamination that may affect analytical results.

8.11.4 Facility Security

The laboratory building is a limited access, secure facility. To ensure that only authorized personnel are able to enter the building from an entrance that is not monitored, entry into each building is limited in one or more of the following ways at a minimum:

- The use of key pads or electronic locks activated by magnetic keys which are issued only to authorized personnel
- Locking doors and issuing keys only to authorized personnel
- Alarm systems to detect unauthorized entrance

During business hours, entry is possible only through the main entrance. This entrance is monitored at all times, usually by a receptionist. All guests are required to sign in by using a visitor logbook.

8.12 Requirements for Ancillary Equipment and Materials

8.12.1 Water

High purity water (e.g., ASTM reagent grade or equivalent water) will be used in all metals, radiological, wet chemistry, and organic analyses. Demonstration of contaminant-free water is shown through the analysis of method blanks consisting of the reagent water on a daily basis for the analyte of interest. This water is obtained by the use of either a commercial ion-exchange deionizing, distillation, or reverse osmosis unit plus an appropriate polishing unit. The resulting water has a maximum conductivity of 1.0 umho-cm at 25°C or a minimum resistivity of 1.0 Mohm at 25°C. Conductivity or resistivity will be monitored and documented daily or on each day that water is dispensed for analytical use.

For volatile analyses the water may be further purified by purging with an inert gas before use to remove potential traces of organic solvents. This is described further in SOP C-MS-0002.

8.12.2 Compressed Air and Gases

Ultra high-purity compressed gases from preapproved vendors or in-house gas generators will be used when required for instrumentation. These air and gases must meet the requirements and specifications of the analytical methods performed. In-line filters will be used when appropriate to minimize contamination and moisture from the gases.

8.12.3 Glassware Preparation

Glassware preparation procedures implemented at operating units are designed to ensure that contaminants are not introduced during sample analysis. Procedures describing glassware preparation are detailed in SOP PITT-QA-0003.

8.12.4 Chemical Storage

Storage of chemicals shall be conducted in a manner to minimize the potential for fire or release of hazardous material resulting from an unplanned chemical reaction. Refrigerators used for storing flammable liquids must have spark-free interior construction. Flammable solvents shall be stored in appropriate cabinets meeting all necessary codes. All chemicals are stored according to chemical compatibility. Further details regarding chemical storage are provided in the CHP.

8.12.5 Waste Management

The goal of STL's policy for waste management is to ensure that laboratory wastes are disposed of safely and in a manner consistent with applicable federal, state and local regulations. The waste disposal program is designed to assure that minimal harm to people and the environment shall result from the disposal of laboratory chemicals. This goal is accomplished by requiring that the laboratory comply with the procedures presented in the CHP.

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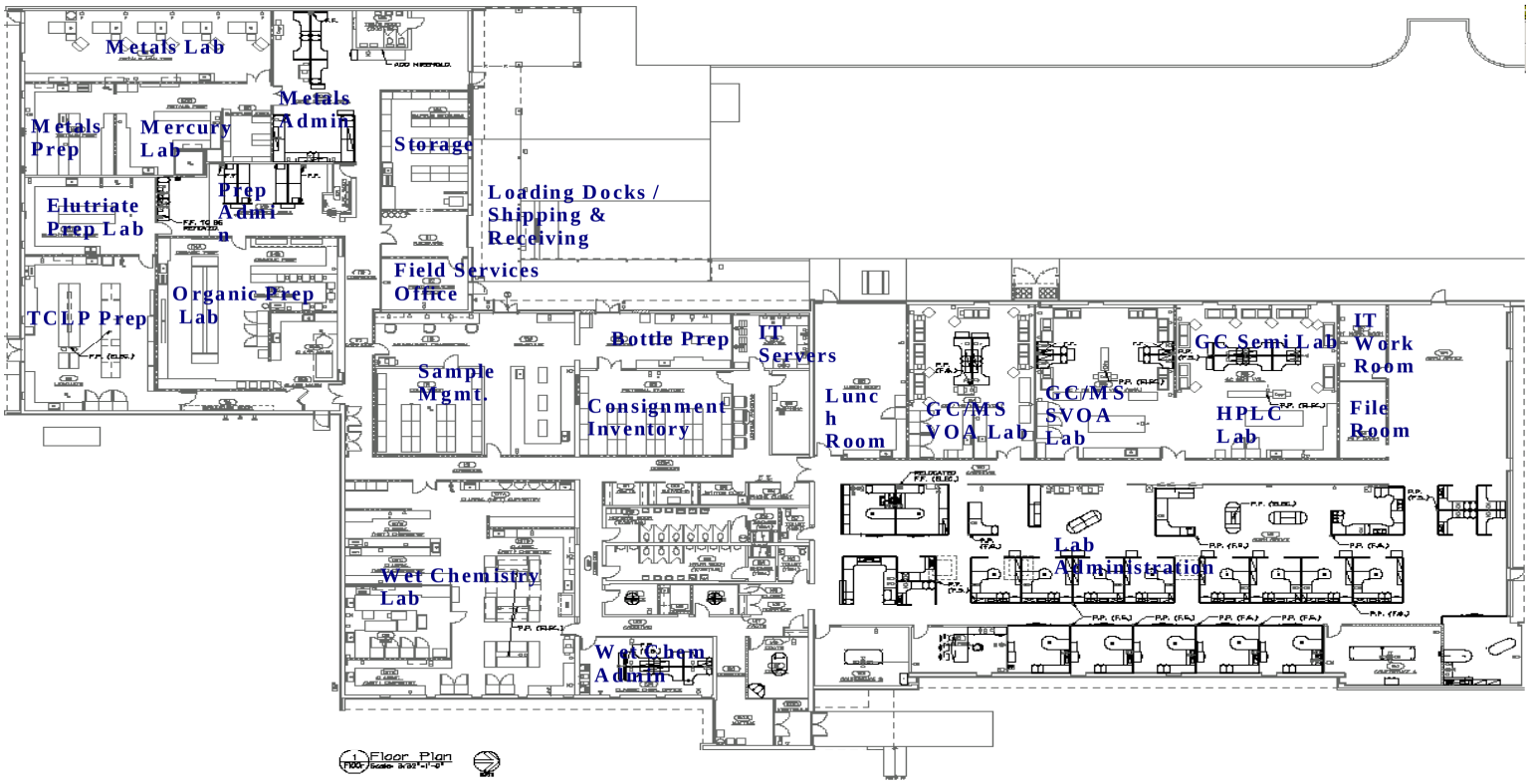
Floor Plan of STL Pittsburgh



Key Areas

- GC/MS VOA (Volatiles)
- GC/MS SVOA (Semivolatiles)
- GC Semi Lab
- HPLC Lab
- Organic Prep Lab
- Wet / General Chemistry
- Metals & Mercury Lab
- Elutriate Lab
- TCLP Prep
- Bottle Prep
- Sample Control/ Sample Receiving
- Field Services

9.0 Quality Assessment and Response

9.1 Nonconformances

A nonconformance is an unplanned deviation from an established protocol or plan and in some cases may be exceptionally permitted departures from the documented policies and procedures or from standard specifications. The deviation may be the result of STL's actions as a systematic error, then termed a deficiency. A single isolated event or event beyond the control of STL is termed an anomaly.

Nonconformances can be identified on the basis of internal or external systems or performance audits, sample processing, routine calibration and monitoring of analytical and support equipment, or QC sample analyses. The Technical Director, Operations Manager, Project Manager, QA Manager, Group Leader, and Analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected, the issue is immediately brought to the attention of QA.

9.1.1 Nonconformance Memo (NCM)

All nonconformances, deficiencies and anomalies, are documented via an electronic process or on a paper form that meets NCM requirements as approved by QA. An allowed exception is log-in conformance problems, which are documented on a Condition Upon Receipt Form (see Section 8.5). A detailed description of the procedure and responsibilities associated with nonconformance documentation, communication, and resolution is described in SOP C-QA-0010, Nonconformance and Corrective Action System.

The Clouseau NCM program, available on the local-area network throughout the laboratory, is the main vehicle for documenting and communicating NCMs. The program allows anyone in the laboratory to document a nonconformance, explain the cause of the problem, and link to the LIMS system to identify the samples and clients involved. The program uses the local e-mail to automatically notify the person's supervisor, the Project Managers associated with the samples, and the QA department. The program is used to document approval and completion of the immediate corrective actions for the samples involved, and can be used to document long-term corrective actions. It provides a place to document resolution of problems with the clients, and it provides routines to query the associated data base to examine trends and prepare management reports. A copy (paper or electronic) of the nonconformance memo will be kept in the project files along with the data it refers to. A copy, paper or electronic, shall also be kept in the quality files.

9.2 Client Complaints

Client inquiries and complaints are generally received through the PM or Customer Services Manager. Typically, the PM or CSM communicates with the client to determine the details of the inquiries, including technical data problems, deliverable issues, turn-around-time problems, etc. Technical and deliverable issues are coordinated by the PM and usually involve input from operations, QA, and management staff. A formal written response to the client is coordinated by the PM, but may on occasion be delivered by the CSM or the Account Manager. Details of the types and levels of complaints and required documentation are provided in STL Corporate SOP S-C-002, Complaint Handling and Service Recovery. Client complaints are recorded in an Excel form, which are summarized in the monthly QA Reports to Management (see Section 9.6 for more about the monthly QA reports).

9.3 Corrective Actions

Corrective actions are measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence. Investigations of potential problems and corrective actions should be timely, determine the root cause, and evaluate any propagation of the error or problem. Whenever a systematic error is discovered that affects the accuracy or defensibility of results reported to STL's clients, Corporate QA involvement followed by written client notification will be part of the corrective action.

Corrective actions should be implemented with an understanding of the technology and work activities associated with the quality element, with appropriate training of STL associates and vendors, and should be monitored for progress and success. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be documented properly. On-the-spot actions are used to correct minor

problems, such as recalibration, retuning, or a minor repair (e.g., replacement of a minor part) of a malfunctioning instrument or the correction of poor analytical technique being used by an analyst. These occurrences are documented in the appropriate injection, run, or analysis logbooks. Similarly, routine instrument maintenance, malfunctions, and power failures are also documented in the appropriate instrument maintenance logbooks. These events do not require a formal NCM process, provided reported analytical results are not affected. Corrective actions specific to quality controls for analytical methods are discussed in the operational-specific SOPs.

9.3.1 Monitoring Corrective Actions

All formal corrective action documentation is maintained by the QA department, either in the Clouseau data base or in paper files. The QA department reviews all corrective actions and selects one or more of the significant corrective actions for inclusion in the annual systems audit. The QA department may also implement a spot assessment audit. The purpose of these audits is to monitor the implementation of the corrective action and to determine whether the action taken has been effective in overcoming the issue identified.

9.4 Internal Audits

Internal audits are performed to assess the degree of adherence to established policies, procedures and standards. These assessments are conducted by STL personnel who are independent of the area being evaluated. Audits can identify areas for improvement with regard to compliance with policies, procedures and standards. Audits also provide a means for correction prior to system failure.

Audits and assessments are generally conducted through the use of checklists and relevant reference documents. The findings of all audits and assessments are documented as is the laboratory response and any corrective actions. Follow-up checks are performed and the status of implementation of corrective actions is documented for all categories of audits and assessments. This cycle continues until all issues are closed.

9.4.1 Audit Types and Frequency

The following types of audits are performed at STL Pittsburgh:

Figure 9.4-1

Audit Types and Frequency

Audit Type	Performed By	Frequency
Systems Audits	QA Department or designee	Annual per lab section
Data Audits	QA Department	5% of all report packages
Spot Assessment	QA Department or designee	As needed to monitor specific issues
Proficiency Testing	Coordinated by QA Dept.	Two samples per year per program as required by NELAC

9.4.2 Systems Audits

Facility systems audits are comprehensive technical and systems evaluations covering each operational and support area at least once per year (see Policy S-Q-002, “Systems Audits”). Generally, a rotating schedule is established throughout the year to ensure adequate coverage of all areas. This schedule can change as situations in the lab warrant. The objectives and schedule of the audit are communicated to the lab groups being assessed in advance of the audit. At the completion of the audit, a debriefing is held to outline the findings, including identification of positive performance, to discuss areas of deficiencies, and to answer questions. The audit report is issued by the QA Manager or their designee, within 30 calendar days of the audit. The audit report is addressed to the area supervisor and/or manager, and copied to the Corporate QA Officer, General Manager, QA Manager (if not the auditor), and Laboratory Director. Written audit responses are required within 30 calendar days of the date of the audit report. The audit response from the lab areas must follow the format of the original audit report, and is sent from the respondents to all individuals copied on the audit report. Where a corrective action requires longer

than 30 days to complete, the target date for the corrective action is stated and evidence of corrective action is submitted to the QA department in the agreed upon time frame.

9.4.3 Data Audits

Data audits are routinely performed and documented to ensure that project records meet project requirements as described in method SOPs, project plans, or other documented requirements. The data audit is used to identify any lab errors that may have occurred. Significant issues found in the course of the audit are brought to the attention of appropriate personnel for clarification, and overseeing correction of final reports if necessary. QA staff are required to perform data audits on 5% of report packages, or more as required by individual national programs. Data audits include spot-checking manual integrations to determine if they are appropriate and documented according to policy S-Q-004. Errors found in client project reports are revised and the revision sent to the client (also see Section 8.9.5).

9.4.4 Spot Assessments

Spot assessments, equivalent to special audits in the STL QMP, are conducted on as needed basis, generally as a follow up to specific issues such as client complaints, validator concerns, corrective actions, control chart or NCM trends, proficiency testing results, data audits, or external audit issues. Spot assessments are focused on a specific issue. The frequency, report format, distribution, and timeframes are tailored to address the nature of the issue.

9.4.5 Proficiency Testing

Proficiency testing samples (PTs) are analyzed to verify the ability of the laboratory to correctly identify and quantitate compounds in PT samples. PT samples may be supplied internally or externally as single-blind or double-blind samples. They can be used to assess if a deficiency has been corrected, they can be used to document the proficiency of the analyst perform the analysis, or they can be used to assess the overall performance of an analytical method.

PT samples are handled and tested in the same manner as environmental samples - it is not acceptable to run multiple replicates that would not otherwise be performed, it is not acceptable to average multiple results, and PT results cannot be shared among labs in advance of the close of the study. PT test sample data is archived using the same requirements as for project and raw data record retention.

9.4.5.1 External PT Samples

STL Pittsburgh participates in a number of PT studies, as shown in Table 9.4-1. The primary one being the NELAC PT program, which involves a minimum of two PT rounds each year for NELAC field of testing for which the lab is maintaining certification.

9.4.5.2 Internal PT Samples

Each STL facility performing chemical analyses also participates in a double-blind performance evaluation annually. An external vendor is contracted to submit double blind samples to the STL labs. Both the level of customer service and the accuracy of the test results is assessed objectively by the external contractor. The PT contractor provides a detailed report to the Corporate QA Manager and to each of the STL facilities.

9.5 External Audits

STL Pittsburgh is regularly audited by clients and external regulatory authorities. STL is available for these audits, and makes every effort to provide the auditors with the personnel, documentation and assistance they require. STL recommends that all audits be scheduled with the QA department so that all necessary personnel are available on the day of the audit. All deficiencies reported to the laboratory must be responded to within the time frame specified by the auditors. It is the responsibility of the QA Manager to coordinate the response to the audit report. The development and implementation of the corrective actions is the responsibility of the operations management of the affected areas. All responses must be approved by the Laboratory Director or Operations Manager prior to submitting the final response. It is the responsibility of the QA Manager to verify implementation of the corrective actions and inform the responsible manager of the closure of all deficiencies from the audit.

9.6 Management Reviews

9.6.1 Quality Reports to Management

A monthly QA report is prepared by the QA Manager and forwarded to the Laboratory Director, the General Manager, and the Corporate QA Manager. The reports include metrics (i.e., frequency and number of revised reports, frequency and number of client complaints) to assess the effectiveness of the Quality System. The contents of the monthly report include:

- Audits

  Results of internal systems audits performed

  Results of external systems audits hosted

  Data audits performed, percent of total packages per month plus any issues

- Revised Reports / Client Complaints

  Frequency of revised reports

  Total number of client complaints, issues, and resolution

- Certification / Parameter Changes

  Proficiency Testing

  Score for each PT as a percentage of maximum score

  Note repeat failures and/or significant problems

- Miscellaneous QA and Operational Issues

Narrative outlining improvements, regulatory compliance issues, general concerns, and assistance required from management.

This information is compiled by the Corporate QA Manager together with similar information from and about other STL laboratories, which is then presented in a report to the STL Chief Operating Officer.

9.6.2 Management Review of QA Systems

The Laboratory Director will conduct annual evaluations the status of the quality systems in the laboratory to review their suitability and effectiveness, and to introduce necessary changes or improvements. The evaluation shall consider:

- The suitability of policies and procedures
- Reports from managerial and supervisory personnel
- The outcome of recent internal audits
- Corrective and preventative actions
- Assessments by external bodies
- The results of interlaboratory comparisons and proficiency tests
- Status of QA documents
- Reviews of QA related requirements in RFPs, SOWs, SAPs, and QAPjPs
- Changes in the volume and type of work and the effects on QA systems
- Client feedback
- Complaints
- Quality control activities
- Resources and staff training

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Specialty Analyses

9.7 Dredged Material Evaluations

STL Pittsburgh offers trace level testing of waters (site-waters and elutriates), sediments, and tissues in support of Dredged Material Evaluations for in-water (ocean and inland waters) and upland (Confined Disposal Facilities (CDFs), beneficial use, etc.) disposal options. In-house capabilities for commonly requested sediment program parameters include:

- Organochlorine Pesticides
- Organophosphorus Pesticides
- PCBs (as Aroclors and Congeners)
- Volatile Organics
- Semivolatile Organics
- Polynuclear Aromatic Hydrocarbons (PAHs)
- Metals
- Cyanide
- Total Sulfides
- Acid Volatile Sulfide (AVS) and Simultaneously Extracted Metals (SEM)
- Nitrogen, Ammonia
- Nitrogen, Nitrate + Nitrite
- Biochemical Oxygen Demand (BOD)
- Chemical Oxygen Demand (COD)
- Total Organic Carbon (combustion procedure for sediments)
- Total Solids/Moisture Content
- Total Volatile Solids
- Lipids

With teaming arrangements with other STL facilities, additional sediment program capabilities include:

- Polychlorinated Dibenzo-Dioxins and Furans (PCDDs/PCDFs)
- Butyl Tins (mono – tetra)
- Total Kjeldahl Nitrogen
- Total Phosphorus
- Grain Size
- Specific Gravity
- Atterberg Limits

STL Pittsburgh also generates elutriate samples following appropriate U.S. Army Corps of Engineers procedures. These include:

- Standard Elutriate Test (SET) for in-water disposal evaluations, and

- Modified Elutriate Test (MET) or Effluent Elutriate Test (EET) for CDF disposal evaluations.
- Illinois Resuspension Tests (Supernatant and Elutriate Tests).
- Dredge Elutriate Test (DRET)

STL Pittsburgh currently supports dredge material evaluation projects following several state specific programs, as well as, under the following guidance documents:

- Ocean Testing Manual or OTM (USACE, 1991).
- New Jersey's Tidal Waters Technical Manual (NJDEP, 1997).
- Inland Testing Manual or ITM (USACE, 1998).
- Upland Testing Manual or UTM (USACE, 2003).

9.8 Tissue Analyses

STL Pittsburgh has extensive experience in supporting projects requiring tissue analyses. These include analyses of laboratory cultured reference species from bioaccumulation tests associated with dredged material evaluations to a variety of field collected species (aquatic and terrestrial). STL Pittsburgh has developed modifications to the standard solid methodologies (where possible) to allow for the use of smaller sample weights and achieve lower quantitation limits. These modifications are supported my tissue specific method performance studies including MDLs in a tissue matrix (the reference tissue is clam). In-house capabilities for commonly requested tissue parameters include:

- Organochlorine Pesticides
- PCBs (as Aroclors and Congeners)
- Semivolatile Organics
- Polynuclear Aromatic Hydrocarbons (PAHs)
- Metals
- Lipids
- Moisture Content

With teaming arrangements with other STL facilities, additional tissue capabilities include:

- Polychlorinated Dibenzo-Dioxins and Furans (PCDDs/PCDFs)
- Butyl Tins (mono – tetra)

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Table Section

2.4-1 LQM Source Documents Requirements Matrix
2.4-2 Cross-Reference of LQM Sections Addressing NELAC Quality Manual Requirements
2.4-3 STL Pittsburgh Quality Documents and Required Approvals
2.4-4 STL Quality Document Review Frequency
3.4-1 STL Pittsburgh Records and Retention Schedule
5.2-1 List of STL Quality-Related Items that Require Evaluation Prior to Use
6-1 GALP Cross Reference to LQM
7.2-1 PADEP List of Certified Parameters
8.0-1 Instrument List
8.2-2 Standard Operating Procedures
8.2-3-1 Wet Chemistry Methods
8.2-3-2 Methods for Mercury by Cold Vapor Atomic Absorption
8.2-3-3 Methods for Metals by ICP and ICPMS
8.2-3-4 Metals Sample Preparation Methods
8.2-3-5 Organic Sample Preparation Methods
8.2-4 Organic Methods RLs
Inorganic Methods RLs
8.4-1 Field Quality Control Samples
8.4-2 Laboratory Quality Control Samples
8.4-3 Laboratory Performance Quality Control Samples
8.4-4 Matrix Specific Quality Control Samples
8.5-1 Inorganic Sample Containers, Preservatives, and Holding Times
8.5-2 Organic Sample Containers, Preservatives, and Holding Times
8.5-3 Sample Containers, Preservatives, and Holding Times for USEPA Contract Laboratory Program Statement of Work
8.5-4 Sample Containers, Preservatives, and Holding Times for TCLP and SPLP
8.5-5 Periodic Equipment Calibrations
8.6-1 Precision and Accuracy Measurements
8.11 Inorganic and Organic Instrument Maintenance Schedules
Wet Chemistry & Misc Instrument Maintenance Schedules
9.4-1 Proficiency Testing Programs
Acronyms and Initialisms
Glossary

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Table 2.4-1

LQM Source Documents Requirements Matrix

STL LQM	EAP QA/R-2	ANSI/ASQC E4-1994	NQA-1[1]	5700.6C[2]	ANSI N 13.30	ANSI/ASQC Q2-1991[3]
1.0 Management Commitment and Organization	1 Management and Organization	2.1 Management and Organization	1 Organization	9.a. General	1.1 Introduction	5.0 Management Responsibility
					1.2 Purpose	
					1.3 Scope	
2.0 Quality System and Description	2 Quality System and Description	2.2 Quality System and Description	2 Quality Assurance Program	1 Program	2.1 Special Word Usage	5.2 Quality System
					2.2 Specific Terms	
					5.1 Quality Assurance	
					5.2 Quality Control	
3.0 Document Control and Records Management	5 Documentation and Records	2.5 Documents and Records	6 Document Control 17 Quality Assurance Records	4 Documents and Records	3.6 Direct Bioassay-Record Retention 4.5 Indirect Bioassay Record Retention	8.4 Quality Documentation and Records

[1] **Section II, "Basic Requirements."**
[2] **Criterion from Section 9, "Requirements."**
[3] **Technically equivalent to ISO9001.**

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Table 2.4-1

LQM Source Documents Requirements Matrix

STL LQM	EAP QA/R-2	ANSI/ASQC E4-1994	NQA-1[1]	5700.6C[2]	ANSI N 13.30	ANSI/ASQC Q2-1991[3]
4.0 Staff Qualification, Orientation and Training	3 Personnel Qualification and Training	2.3 Personnel Training and Qualification		2 Personnel Training and Qualification	3.2 Personnel Preparation	14.0 Personnel
5.0 Procurement of Supplies and Services	4 Procurement of Items and Services	2.4 Procurement of Items and Services	4 Procurement Document Control 7 Control of Purchased Items and Services	7 Procurement	N/A	7.0 Quality in Procurement 13.0 Subcontracting
6.0 Computer Hardware and Software	6 Computer Hardware and Software	2.6 Computer Hardware and Software	3 Design Control	N/A	N/A	ISO 9000-3[4]
7.0 Contract Review and Project Planning	7 Planning	2.7 Planning	2 Quality Assurance Program	1 Program	3.1 Facility Criteria	6.3.3 Quality Plans
		3.1 Planning and Scoping	3 Design Control	6 Design	3.4 Direct Bioassay – Performance Criteria for Services Laboratories	

[1] **Section II, "Basic Requirements."**
[2] **Criterion from Section 9, "Requirements."**
[3] **Technically equivalent to ISO9001.**
[4] **Quality Management and Quality Assurance Standards, ISO 9000, Part 3, "Guidelines for the Application of ISO 9001 to the Development, Supply and Maintenance of Software."**

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Table 2.4-1

LQM Source Documents Requirements Matrix

STL LQM	EAP QA/R-2	ANSI/ASQC E4-1994	NQA-1[1]	5700.6C[2]	ANSI N 13.30	ANSI/ASQC Q2-1991[3]
		3.3 Implementation of Planned Operations	5 Instructions, Procedures, and Drawings	N/A	3.5 Direct Bioassay – Reporting Results	
			9 Control Processes		4.2 Indirect Bioassay – Analytical Methodology	
			11 Test Control		4.3 Indirect Bioassay – Performance Criteria for Service Laboratories	
			13 Handling, Storage, and Shipping		5.2 Quality Control	

[1] **Section II, "Basic Requirements."**
[2] **Criterion from Section 9, "Requirements."**
[3] **Technically equivalent to ISO9001.**

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Table 2.4-1

LQM Source Documents Requirements Matrix

STL LQM	EAP QA/R-2	ANSI/ASQC E4-1994	NQA-1[1]	5700.6C[2]	ANSI N 13.30	ANSI/ASQC Q2-1991[3]
8.0 Work Processes and Operations	8 Implementation of Work Processes	2.8 Implementation of Work Processes	1 Organization	5 Work Processes	3.1 Facility Criteria	8.0 Laboratory Operations Quality Assurance
			5 Instructions, Procedures, and Drawings	6 Design		9.0 Control of Measuring and Test Equipment 10.0 Data Validation 15.0 Use of Statistical Methods
	8 Implementation of Work Processes (Continued)	3.2 Design of Data Collection Operations	10 Inspection 12 Control Measuring and Test Equipment	8 Inspection and Acceptance Testing		
			14 Inspection, Test, and Operating Status			
9.0 Quality Assessment and Response	9 Assessment and Response[4]	2.9 Assessment and Response	2 Quality Assurance Program	9 Management Assessment	3.3 Direct Bioassay – Interpretation of Measurements	16.0 Nonconformity

[1] **Section II, "Basic Requirements."**
[2] **Criterion from Section 9, "Requirements."**
[3] **Technically equivalent to ISO9001.**
[4] **This document has two sections numbered "9."**

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Table 2.4-1

LQM Source Documents Requirements Matrix

STL LQM	EAP QA/R-2	ANSI/ASQC E4-1994	NQA-1[1]	5700.6C[2]	ANSI N 13.30	ANSI/ASQC Q2-1991[3]
		3.4 Assessment and Response	13 Handling, Storage, and Shipping		3.5 Direct Bioassay – Reporting Results	17.0 Corrective Action
		3.5 Assessment and Verification of Data Usability	15 Control of Nonconforming Items	10 Independent Assessment	4.4 Indirect Bioassay – Reporting Results	18.0 Auditing the Quality System
			16 Corrective Action		6.1 Direct Bioassay Measurements	
			18 Audits		6.2 Indirect Bioassay Measurements	
N/A	9 Quality Improvement 5	2.10 Quality Improvement	N/A	3 Quality Improvement	N/A	N/A

[1] **Section II, "Basic Requirements."**
[2] **Criterion from Section 9, "Requirements."**
[3] **Technically equivalent to ISO9001.**

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Table 2.4-2

Cross-Reference of LQM to NELAC Requirements for Quality Manuals

NELAC QUALITY MANUAL: REQUIRED ELEMENTS[1]	LQM, QA POLICY, AND/OR QA SOP REFERENCE
1. A quality policy statement, including objectives and commitments by top management	LQM Chapter 1
2. The organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts	LQM Chapter 1
3. The relationship between management, technical operations, support services and the quality system	LQM Chapter 1 LQM Chapter 7
4. Procedures to ensure that all records required under this chapter are retained as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force	LQM Chapter 2 LQM Chapter 3
5. Job descriptions of key staff and reference to the job descriptions of other staff	LQM Chapter 1 and Chapter 4 Separate document (hard copy and/or electronic) provides job descriptions
6. Identification of the laboratory's approved signatories; at a minimum, the title page of the Quality Manual must have the signed concurrence, (with appropriate titles) of all responsible parties including the QA officer, technical director, and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager	LQM Title/Approved Page
7. The laboratory's procedures for achieving traceability of measurements	LQM Chapter 8
8. A list of all test methods under which the laboratory performs its accredited testing	LQM Table 8.2-1
9. Mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	LQM Chapter 7

[1] **National Environmental Laboratory Accreditation Conference Standard, Quality Systems, July 1, 1999**

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Table 2.4-2

Cross-Reference of LQM to NELAC Requirements for Quality Manuals

NELAC QUALITY MANUAL: REQUIRED ELEMENTS[1]	LQM, QA POLICY, AND/OR QA SOP REFERENCE
10. Reference to the calibration and/or verification test procedures used	LQM Chapter 8 Tables 8.5-6 through 8.5-7
11. Procedures for handling submitted samples	LQM Sections 8.5.2 and 8.5.3
12. Reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests	Equipment list is Table 8.0-1 LQM Section 5.2.4, 8.1, 8.5.4, 8.11 to 8.12
13. Reference to procedures for calibration, verification and maintenance of equipment	Calibrations in Tables 8.5-6 through 8.11-30 LQM Sections 8.5.4, 8.11 to 8.12
14. Reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes	LQM Section 9.4.5
15. Procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur	LQM Chapter 9 SOP C-QA-0010, “Nonconformance and Corrective Action”
16. The laboratory management arrangements for exceptionally permitting departures for documented policies and procedures or from standard specifications	LQM Section 7.4 and 9.1
17. Procedures for dealing with complaints	LQM Section 9.2
18. Procedures for protecting confidentiality and proprietary rights (including national security concerns)	LQM Section 8.9.6
19. Procedures for audits and data reviews	LQM Section 5.3.6, 9.8, and 9.4 SOP C-QA-0004 “Independent QA Data Review”
20. Processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and/or receive any needed training	LQM Chapter 4 SOP C-QA-0013, “Employee Orientation Training”

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Table 2.4-2

Cross-Reference of LQM to NELAC Requirements for Quality Manuals

NELAC QUALITY MANUAL: REQUIRED ELEMENTS[1]	LQM, QA POLICY, AND/OR QA SOP REFERENCE
21. Process/procedures for educating and training personnel in their ethical and legal responsibilities including the potential punishment and penalties for improper, unethical or illegal actions	LQM Section 1.4 Policy QA-008 – Data Recording Requirements QA-010 – Maintaining Timely Integrity S-!-004 – Acceptable Manual Integration Practices P-T-001 – Selection of Data Points Required for Initial Calib.
22. Reference to procedure for reporting analytical results	LQM Section 8.9 Policy QA-004, “Rounding and Significant Figures” Policy QA-009, “Reporting Limits”
23. A table of contents and applicable list of references, glossaries, and appendices	LQM Table of Contents List of Policies and SOPs Table 2.3-2

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Table 2.4-3

STL Pittsburgh Quality Documents and Required Approval

Quality Document	Required Approvals
Laboratory Quality Manual (LQM)	Laboratory Director Technical Director Quality Assurance Manager
STL Pittsburgh Policies	Laboratory Director Quality Assurance Manager
STL Pittsburgh Standard Operating Procedures (SOPs)	Laboratory Director Technical Specialist Laboratory Health and Safety Coordinator[1] Quality Assurance Manager

[1] **Required only if procedure encompasses more than standard office safety requirements.**

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Table 2.4-4

STL Quality Document Review Frequency

Document Type	Frequency of Review	Responsible Party
Laboratory Quality Manual (LQM)	Annual	Quality Assurance Manager
Standard Operating Procedures (SOPs)	Annual	Quality Assurance Manager and Operations Manager

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Table 3.4-1

STL Pittsburgh Records and Retention Schedule

Type of Record	Retention	Disposition
General Laboratory Documents		
Instrument output	7 yrs from project completion	Destroy
Quality control data	7 yrs from project completion	Destroy
Field sample date	7 yrs from project completion	Destroy
Final analytical reports	7 yrs from project completion	Destroy
Instrument logbooks	7 yrs from last entry	Destroy
Equipment monitoring and maintenance records	7 yrs from last entry	Destroy
Instrument calibration records	7 yrs from last entry	Destroy
Standard preparation logs	7 yrs from last entry	Destroy
Standards certificates	7 yrs from last entry	Destroy
Measurement and test equipment logs (e.g., refrig., balances, etc.)	7 yrs from last entry	Destroy
Method and instrument validation records	7 yrs from last entry	Destroy
Instrument manuals	Retain until superseded	Destroy
Project management files	7 yrs from date of archival	Destroy
Quotes and proposals	2 yrs from date of expiration	Destroy
LQM, policies, and SOPs	Indefinite review every 3 yrs	Destroy
Analyst demonstration of proficiency	5 yrs from date of archival	Destroy
Quality assurance audits	5 yrs from last entry	Destroy
Certifications and approvals	Indefinite review every 3 yrs	Destroy
Employee signature list	7 yrs from date of archival	Destroy
MDL studies	7 yrs from last entry	Destroy
Performance testing studies	7 yrs from last entry	Destroy
QA reports to management	7 yrs from last entry	Destroy
Quality control charts	7 yrs from last entry	Destroy
Environment, Health and Safety Records		
Medical records	Retain while active and 30 years from last entry	Destroy
Employee exposure and monitoring records	Retain while active and 30 years from last entry	Destroy
Workers compensation files and first report of injury	Retain while active and 18 years from last entry	Destroy
Accident logs (OSHA Form 200)	7 yrs from last entry	Destroy
Accident reports	7 yrs from last entry	Destroy
Environmental permits	7 yrs from last entry	Destroy
Environmental management, e.g., discharge reports	5 yrs from last entry	Destroy
Health and safety audits	7 yrs from last entry	Destroy

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Table 3.4-1

STL Pittsburgh Records and Retention Schedule

Type of Record	Retention	Disposition
Chemical Hygiene Plan	5 yrs from archival	
Safety Inspections	5 yrs from last entry	Destroy
Radioactive materials records	7 yrs from last entry	Destroy
NRC or state radioactive materials handling inspections	5 yrs from last entry	Destroy
TLD exposure records	5 yrs from last entry	Destroy
EH&S training	7 yrs from last entry	Destroy
Accounting	See Accounting and Controls Procedures Manual	
Administrative		
Personnel records (not including medical or disability records)	7 yrs from last entry	Destroy

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Table 5.2-1

List of STL Quality-Related Items that Require Evaluation Prior to Use

Quality-Related Item	Standard Operating Procedures for Quality Testing
Acetone	S-T-001
Dichloromethane	S-T-001
Hexane	S-T-001
Hydrochloric acid	S-T-001
Freon	S-T-001
Methanol	S-T-001
Nitric acid	S-T-001
Hydrogen Peroxide	S-T-001
Sulfuric acid	S-T-001
Toluene	S-T-001

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Table 6-1

GALP Cross Reference to LQM

GALP Section	GALP Guidance	STL Document
8.1 Laboratory Management	8.1.1 ensure that personnel clearly understand the functions they are to perform	LQM 1.6.2, 1.6.4, and 4.0
	8.1.2 ensure that QAU monitors computer activities	LQM 9.4.2
	8.1.3 ensure that personnel, resources, and facilities are adequate and available as scheduled	LQM 1.6.1 - 1.6.4
	8.1.4 receive reports of QAU inspection and audit reports and ensure corrective actions are promptly taken in response to any deficiencies	LQM 9.2.2.1
	8.1.5 approve SOPs related to the computer activities, and ensure that deviations to the SOPs are documented	LQM 3.3 and 9.1.4
	8.1.6 assure that GALP provisions are followed	LQM 6.0
8.2 Personnel	8.2.1 must have adequate education, training, and experience to perform assigned IT functions	LQM 4.0
	8.2.2 a summary of training, experience, and job description must be maintained	LQM 4.1
	8.2.3 personnel must be of a sufficient number for timely and proper operation of the computer systems	LQM 1.6.2
8.3 Quality Assurance Personnel	8.3.1 shall be separate and independent of IT personnel, and shall report directly the laboratory management	LQM 1.6.1
	8.3.2 shall have immediate access to the computer data, SOPs, and other records	LQM 1.6.1
	8.3.3 inspect the LIMS at intervals to ensure the integrity of LIMS raw data, and shall present inspection reports to management	LQM 9.4.2 and 9.4.3

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Table 6-1

GALP Cross Reference to LQM

GALP Section	GALP Guidance	STL Document
	8.3.4 determine that no deviations from approved SOPs were made without proper authorization and documentation	LQM 9.1.1
	8.3.5 periodically audit raw data to ensure their integrity	LQM 9.4.4 and 9.4.3
	8.3.6 maintain adequate records of all QAU operations	LQM 9.4.4 and 9.4.3
8.4 LIMS Raw Data	8.4.1 LIMS raw data and the storage media on which they reside must be identified and documented. The documentation shall be included in the lab's SOPs.	System map is with IS Director
	8.4.2 the individual(s) responsible for entering and recording LIMS raw data must be uniquely identified, together with the date and time the data were entered	QA-008
	8.4.3 the instrument transmitting raw data must be uniquely identified in the record, together with the date and time of transmission	QA-008
	8.4.4 procedures and practices used to verify LIMS raw data must be documented in controlled SOPs	CORP-IT-007 LQM 8.8 – 8.8.3
8.5 Software	8.5.1 SOPs shall be established for software development, software testing, change control, version control, maintaining historical file	P-ITQ-013 S-ITQ-0001 S-ITQ-0007
	8.5.2 documentation shall be maintained for software description and functional requirements,	P-ITQ-013

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Table 6-1

GALP Cross Reference to LQM

GALP Section	GALP Guidance	STL Document
	algorithms and formulas, testing and quality assurance	
	8.5.3 all documentation is readily available in the facility where the software is used and SOPs are readily available where procedures are performed	P-PTQ-013
	8.5.4 a historical fail of software and documentation shall be retained	S-ITQ-0001, Sect 4.14.1
8.6 Security	Laboratory management shall ensure that security practices are adequate to assure the integrity of data	LQM 6.2 P-ITQ-013
8.7 Hardware	8.7.1 must be of adequate design and capacity and a documented description maintained	LQM 6.1 P-PTQ-013
	8.7.2 must be installed in accordance with manufacturer's recommendations, and undergo documented acceptance testing as described in a laboratory SOP	S-ITQ-001 P-ITQ-013 LQM 6.1
	8.7.3 testing, maintenance and repair must be described in a laboratory SOP	P-ITQ-013
8.8 Comprehensive Testing	Management shall ensure that comprehensive testing shall be documented at least every 24 months or more frequently as a result of software changes	S-ITQ-0001 LQM 6.3.4
8.9 Records Retention	Procedures must be in place for the retention of LIMS raw data and documentation and records pertaining to LIMS	LQM 3.4 – 3.5
8.10 Facilities	8.10.1 the environmental conditions of the facility housing the LIMS must be controlled to protect against	LQM 6.2

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Table 6-1

GALP Cross Reference to LQM

GALP Section	GALP Guidance	STL Document
	data loss	
	8.10.2 environmental conditions for storing LIMS raw data and records must be adequate	LQM 6.2
8.11 SOPs	8.11.1 SOPs, as described above, must be maintained and readily available where the procedure is performed	LQM 3.1 – 3.2 SOP Index
	8.11.2 SOPs must be reviewed periodically to ensure that they are accurate	LQM 3.3
	8.11.3 SOPs must be authorized and controlled, with all changes subject to the same approvals and control	LQM 3.3
	8.11.4 an historical file of SOPs must be maintained	

Table 7.2-1

PADEP List of Certified Parameters (See current cert for detailed list)

Parameter	Method	Matrix	Certified[1]
VOAs	8260B	Water	Yes
VOAs	8260B	Soil	Yes
BNAs	8270C	Water	Yes
BNAs	8270C	Soil	Yes
PCBs	8082	Water	Yes
PCBs	8082	Soil	Yes
PCB Congeners	8082	Water/Soil	Yes
Pesticides	8081A	Water	Yes
Pesticides	8081A	Soil	Yes
PAHs	8310	Water	Yes
PAHs	8310	Soil	Yes
EDB/DBCP	8011	Water	Yes
OPPs	8141A	Water	Yes
OPPs	8141A	Soil	Yes
Herbicides	8151A	Water	Yes
Herbicides	8151A	Soil	Yes
ICP Trace	200.7	Water	Yes
ICPMS	200.8	Water	Yes
ICP Trace	6010B	Water	Yes

[1] **Copy of current certificate which includes all methods and analytes is available upon request**

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Table 7.2-1

PADEP List of Certified Parameters (See current cert for detailed list)

Parameter	Method	Matrix	Certified[1]
ICP Trace	6010B	Soil	Yes
ICPMS	6020	Water	Yes
ICPMS	6020	Soil	Yes
ICPMS	6800	Water/Soil	Yes
Hg	245.1	Water	Yes
Hg	7470A	Water	Yes
Hg	7471A	Soil	Yes
Color	110.2	Water	Yes
Conductivity	120.1	Water	Yes
Hardness	130.2, 2340B	Water	Yes
Total Hardness as $CaCO_3$	2340B, C	Water	Yes
pH	150.1	Water	Yes
TDS (residue filterable)	160.1	Water	Yes
TSS (residue nonfilterable)	160.2	Water	Yes
TS (total residue)	160.3	Water	Yes
Volatile Residue	160.4	Water	Yes
Residue Settleable	160.5		
Oil & Grease (HEM-Water)	1664A	Water	Yes
TPH, Recoverable (SGT-HEM-NON POLAR MATERIAL)	1664A	Water	Yes
Oil and Grease	9070	Water	Yes
Oil an Grease	9071	Soil	Yes
Chloride	300.0	Water	Yes
Nitrate	300.0/353.2	Water	Yes
Nitrite	300.0/353/2	Water	Yes
Orthophosphate as P	300.0	Water	Yes
Sulfate	300.0	Water	Yes
Bromide	300.0	Water	Yes
Fluoride	300.0	Water	Yes
Fluoride	340.2	Water	Yes
Acidity as CaCO3	305.1/2310B	Water	Yes
Alkalinity	E 310.1	Water	Yes
Alkalinity	SM 2320B	Water	Yes
Alkalinity	310.2 (Autotitration)	Water	Yes
Chloride	E325.2,SM 4500ClE	Water	Yes
Cyanide, total (midi distillation)	E335.4, 9010	Water	Yes
Cyanide, total (automated)	9012	Water	Yes
Cyanide Extraction of Soils and Oils	9013	Soil/Oil	Yes
Available Cyanide	OIA 1677	Water/Soil	Yes
Ammonia	E350.1	Water	Yes

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Table 7.2-1

PADEP List of Certified Parameters (See current cert for detailed list)

Parameter	Method	Matrix	Certified[1]
Nitrate as N	E353.2,SM 4500NO_3F	Water	Yes
Sulfate - Turbidimetric	E 375.4	Water	Yes
Sulfide	E 376.1	Water	Yes
BOD, 5 day	E 405.1	Water	Yes
BOD, 5 day	SM 5210B	Water	Yes
COD	410.4	Water	Yes
COD	Hach 8000	Water	Yes
TOC	415.1	Water	Yes
Phenolics, total	420.2	Water	Yes
Chromium VI	SM 3500 - Cr B and D	Water	Yes
Chromium VI	7196A	Water	Yes
Chromium VI	7196A	Soil	Yes
Flashpoint (Ignitability)	1010	Water/Soil	Yes
Ignitability	1020A	Waste	Yes
Sulfide	9030B(dist) 9034(titra)	Water	Yes
Sulfide	9030B(dist) 9034(titra)	Soil	Yes
pH	9040B	Water	Yes
pH	9045B & C	Soil	Yes
Chloride	9056 (IC)	Water	Yes
Fluoride	9056 (IC)	Water	Yes
Nitrate	9056 (IC)	Water	Yes
Nitrite	9056 (IC)	Water	Yes
Orthophosphate as P	9056 (IC)	Water	Yes
Sulfate	9056 (IC)	Water	Yes
TOC	9060	Water	Yes
Total Phenolics	9066/9065	Water	Yes
Paint Filter Liquid Test	9095	Water	Yes

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Table 8.0-1

Instrument List

Instrument Type	Manufacturer	Model	Purchase Date	Autosampler
GC				
	Hewlett-Packard S/N 3235A48356	5890II Dual ECD with EPC	1991	Yes
	Hewlett-Packard S/N 3118A35332	5890II Dual ECD	1989	Yes
	Hewlett-Packard S/N 2950A27000	5890II Dual ECD	2001	Yes
	Hewlett-Packard S/N US00024872	6890 Dual ECD	1998	Yes
	Hewlett-Packard S/N US00023401	6890 Dual ECD	1998	Yes
	Hewlett-Packard S/N US10237038	6890 Dual ECD	2002	Yes
	Hewlett-Packard S/N US00025516	6890 Dual NPD	1998	Yes
	Hewlett-Packard S/N US10145113	6890 Dual FPD	2001	Yes
	Hewlett-Packard S/N US1014S114	6890 Dual ECD	2001	Yes
	Hewlett-Packard S/N 226398	6890 Dual ECD	2005	Yes
	Hewlett-Packard S/N US10403014	6890 Dual ECD	2006	Yes
GC/MS Volatiles	Hewlett-Packard S/N US00010799(GC) S/N US72821085(MSD)	6890-GC 5973-MSD	1998	Yes Archon
	OI Eclipse Concentrator D616466032P		2006	NA
	Hewlett-Packard S/N US00009844(GC) S/N US72020964(MSD)	6890-GC 5973-MSD	1997	Yes Archon
	OI Eclipse Concentrator D617466100P		2006	NA
	Hewlett-Packard S/N US00001295-(GC) S/N3526I01420-(Headspace)	6890 FID	2001	Yes HP7694
	Hewlett-Packard S/N US00023292(GC) S/N US82322212(MSD)	6890-GC 5973-MSD	1998	Yes Archon
	OI Eclipse Concentrator D616466026P		2006	NA
	Hewlett-Packard	6890-GC	1999	Yes

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Table 8.0-1

Instrument List

Instrument Type	Manufacturer	Model	Purchase Date	Autosampler
	S/N US00030465(GC) S/N US92522786(MSD)	5973-MSD		Archon
	OI Eclipse Concentrator B414466952P		2006	NA
GC/MS Semivolatiles	Hewlett-Packard S/N US00029391 (GC) S/N US9142251(MSD)	6890-GC 5973-MSD	1999	Yes HP7683
	Hewlett-Packard S/N US00029396 (GC) S/N US91922512(MSD)	6890-GC 5973-MSD	1999	Yes HP7683
	Hewlett-Packard S/N CN10426047 (GC) S/N US41746674 (MSD)	6890-GC 5973-MSD	2004	Yes HP7683
	Hewlett-Packard S/N US00031329 (GC) S/N US93112052(MSD)	5890-GC 5972-MSD	2000	Yes
	Hewlett-Packard S/N US91411735	5890-GC 5972-MSD		Yes
	Hewlett-Packard S/N US 71410457	5890-GC 5972-MSD		Yes
	Hewlett-Packard S/N US80210935	5890-GC 5972-MSD		Yes
HPLC	Hewlett-Packard S/N (per component)	1100 UV and Fluorescence	1998	Yes
ICP	Thermo Jarrell Ash S/N 38190	61E ICAP	1992	Yes
	Thermo Jarrell Ash S/N 209390	61E Trace	1993	Yes
	Thermo Jarrell Ash S/N 11097	61E Trace	2001	Yes
ICP/MS	Thermo Electron	X-Series ICPMS	2003	Yes
ICP/MS	Thermo Electron	X Series ICPMS	2006	Yes
IC	Metrohm	709 IC Pump Autosampler 838	2006	Yes
Mercury Analyzer	Leeman Labs S/N 3009	Hydra	2003	Yes
	Leeman Labs S/N HG9007	PS 200II	1999	Yes
Ion Chromatograph	Dionex S/N 00040396	IC 25	2000	Yes
Autoanalyzer	Alpkem	Flow Solution IV	1998	Yes

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Table 8.0-1

Instrument List

Instrument Type	Manufacturer	Model	Purchase Date	Autosampler
	S/N 928893439			
	Alpkem S/N 928893438	Flow Solution IV	1998	Yes
GPC	J2 Scientific S/N 084/12298	Autoinject 110	2001	Yes
UV/VIS	Beckman Coulter S/N 4325089	DU640	2000	No
	Milton Roy S/N 3V08239002	Genesys5	2003	No
	Milton Roy S/N 3155215007	SPEC-21D	1994	No
Midi Distillers	Westco Scientific S/N 1064	Easy Dist	2000	No
	Westco Scientific S/N 1064	Easy Dist	2000	No
	Westco Scientific S/N 1081	Easy Dist	2001	No
pH meter	Fisher Scientific S/N AR93315378	AR25	2004	No
	Fisher Scientific S/N AR93312320	AR25	1990	No
	Fisher Scientific S/N AR 81202030	AR25	2003	No
Conductance Meter	YSI S/N 705	32	1985	No
COD Reactor	Hach S/N 1131194	DRB200	2005	No
	HACH S/N 020300022933	45600	2002	No
TOC	OI Analaytical S/N 5108710555	Model# 1010	2001	Yes
TOC (Lloyd Khan Method)	Thermo Electron Corp. Flash EA 112 MAS 200R NC Soil Analyzer	20057159-20057135	2006	Yes
Konelab 20	Thermo Clinical Labsystems	Aqua 200	2005	Yes
1677 Autoanalyzer	OI Analytical S/N 135804017	Model# A0001604	2001	Yes
BOD Meter	YSI S/N 03L0794	52	2004	Yes
	YSI S/N 91K033593	50B	2003	No
Sonicator	Heat Systems	Ultrasonic	1985	No

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Table 8.0-1

Instrument List

Instrument Type	Manufacturer	Model	Purchase Date	Autosampler
	S/N G1026	Processor XL		
	Fisher Scientific	550 Sonic Dismembrator	1985	No
Soxtherms	Gerhardt S/N 4012404	SE-3A/5306A	2002	No
	Gerhardt S/N 4012399	SE-3A/5306A	2002	No
	Gerhardt S/N	SE-3A/5306A	2002	No
	Gerhardt S/N 4012398	SE-3A/5306A	2002	No
	Gerhardt S/N 4012403	SE-3A/5306A	2002	No
	Gerhardt S/N 4012402	SE-3A/5306A	2002	No
	Gerhardt S/N 4012401	SE-3A/5306A	2002	No
	Gerhardt S/N 4012400	SE-3A/5306A	2002	No
Flashpoint	Rapid Tester S/N 024149	RT-00001	2002	No
	Petrotest Pensky Martin S/N 0741043006	PMA-4	2004	No

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Table 8.2-2

Standard Operating Procedures

SOP No.	Title	Rev. No.	Rev. Date
S-ITQ-0001	Change Management	1	2/5/2003
S-ITQ-0005	LIMS user profile setup and maintenance	1	2/5/2003
S-ITQ-0007	Software Testing, Validation and Verification	1	1/27/2003
P-AR-001	Revenue Recognition	0	12/13/2002
P-AR-002	Bad Debt Allowance and Write Off	0	4/1/2002
PC-01	Petty Cash Policy	0	9/20/2002
P-CR-001	Establishing Customer Credit	0	12/16/2002
P-CR-002	Collection	0	12/16/2002
P-E-001	Crisis Management Policy	2	2/28/2002
P-E-002	Annual Assessment and Action Plan Preparation	1	1/1/2003
P-I-001	Internet Use Policy	2	5/25/2004
P-I-002	Electronic Mail	2	5/25/2004
P-I-003	Account and Naming	2	5/25/2004
P-I-004	Password Policy	2	5/25/2004
P-I-005	Software Licensing Policy	2	10/11/2004
P-I-006	Virus Protection Policy	2	10/11/2004
P-I-007	Data Backup Policy	2	10/11/2004
P-I-008	Internet Security Policy	2	1/20/2004
P-I-009	Cellular Telephone Policy	1	6/14/2004
P-I-010	VPN Network Access Policy	1	9/22/2006
P-I-011	Business Partner Network Access Policy	1	9/22/2006
P-I-012	Contractor Network Access Policy	1	9/22/2006
P-I-013	Internet Access Policy	1	9/22/2006
P-I-014	Network Access Policy	1	9/22/2006
P-I-015	LAN Network Architecture	1	9/22/2006
P-L-001	Record Retention Policy	1	9/17/2004
P-L-002	Subpoenas Policy	2	4/28/2004
P-L-003	Internal Investigations Policy	1.1	2/3/2005
P-L-004	Organizational Conflicts of Interest	1	9/17/2004
P-L-006	Ethics Policy	4.1	2/3/2005
P-ITQ-013	Software Quality Assurance	1	1/27/2003
P-PU-001	Purchase Order Requirements	0	9/1/2002
P-PU-002	Authorization Matrix	3	7/1/2002

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Table 8.2-2

Standard Operating Procedures

SOP No.	Title	Rev. No.	Rev. Date
P-PU-003	CapEx and Lease Approval	0	11/1/2002
P-T-001	Selection of Calibration Points	3	9/8/2004
P-T-002	Establishment and Utilization of Technical Experts	1	4/11/2001
P-T-003	Qualified Product List	1	8/6/2002
S-AP-001	Invoice Processing	0	12/13/2002
S-AR-002	Cash Application	0	12/13/2002
S-C-001	Work Sharing Process	1	10/11/2004
S-C-002	Complaint Handling and Service Recovery	1	7/21/2006
S-E-001	Procedures for Shipping Samples and Kits	2	5/16/2001
S-F-009	Purchase Order Requirements	1	10/4/2006
S-PU-001	Requisition of Sub-Contract Services	0	5/21/2003
S-PU-002	Returns Vendor Performance	0	12/31/2002
S-PU-003	Rush Emergency Orders	0	12/31/2002
S-Q-001	Official Document Control and Archive	5	1/2/2007
S-Q-002	Systems Audits	3.1	2/3/2005
S-Q-003	MDL Policy	2	11/24/2004
S-Q-004	Manual Integration	3	1/2/2007
S-Q-005	Data Recall Process	1.1	2/3/2005
S-Q-007	Data Authenticity Audits	1	2/3/2005
S-T-001	Testing of Solvents and Acids	2	12/7/2004
S-T-002	Reporting Limits for QuantIMS	2	12/7/2004
STL QMP	**STL Quality Management Plan**	**7**	**11/4/2005**
C-GC-0001	Chromatographic Analysis Based on Method 8000B, SW-846 8081A, 8082, 8141A, 8151A, 8310 and 8041	9	10/21/05
PITT-GC-0042	Extraction and Analysis of Chlorinated Pesticides and PCBs by OLM04.2	0	09/30/99
PITT-GC-8011	1,2-Dibromoethane(EDB) and 1,2-Dibromo-3-Chloropropane(DBCP) in Water by Microextraction and Gas Chromatography, Method 8011	3	10/20/05
PITT-GC-W-0001	Work Instruction for GC/HPLC Methods Summary of Calibration Verification Criteria	0	12/15/05
PITT-HS-0002	Waste Characterization and Categorization	2	08/29/06
PITT-HS-0003	Waste Collection, accumulation and Storage	3	10/01/06

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Table 8.2-2

Standard Operating Procedures

SOP No.	Title	Rev. No.	Rev. Date
PITT-HS-0004	Waste Shipping and Manifesting	2	08/29/06
PITT-HS-0005	Sample Disposal	1	04/01/03
C-IP-0002	Acid Digestion of Soils, SW-846 Method 3050B	5	09/28/06
C-IP-0003	Acid Digestion of Aqueous Samples by SW-846 and MCAWW 200 Series Methods	5	09/28/06
C-IP-0004	Toxicity Characteristic Leaching Procedure and Synthetic Precipitation Leaching Procedure	2	04/01/03
PITT-IP-0001	Acid Digestion of Waters and Soils, CLP SOW ILM03.0 & 4.0	3	09/28/06
PITT-IP-0022	Extraction Procedure Test for Plant Bioaccumulation - DTPA Extraction Procedure	1	10/05/06
PITT-IP-9013	Method SW-9013 Cyanide Extraction Procedure for Solids and Oils	1	04/29/05
SW846 Method 1320	Multiple Extraction Procedure	0	09/01/86
WI-IT-0001	Work Instruction for Servers Data Back-up	2	05/01/06
C-MS-0001	GCMS Analysis Based on Method 8270C	6	10/17/05
C-MS-0002	Volatile Organics by GC/MS Based on Methods 8260B	8	10/24/05
PITT-MS-0003	Analysis of Polynuclear Aromatic Hydrocarbons by Selective Ion Monitoring	2	10/19/05
PITT-MS-B042	Extraction and Analysis of Semivolatiles (BNAs) by EPA CLP OLM04.2	0	09/30/99
PITT-MS-V042	GCMS Volatile Organic Analysis by EPA CLP SOW OLM04.2	0	09/30/99
PITT-MS-WI-0010	SW-846 8270C Calibration Criteria and Corrective Actions Appendix B for C-MS-0001	1	04/28/05
C-MT-0001	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW-846 Method 6010B and EPA Method 200.7	7	10/17/05
C-MT-0005	Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCAWW 245.1	5	10/17/05
C-MT-0007	Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, SW846 7471A and MCAWW 245.5	3	04/01/03
PITT-MET-WI-0001	Metals Prep Guide - STL Pittsburgh	1	04/26/05

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Table 8.2-2

Standard Operating Procedures

SOP No.	Title	Rev. No.	Rev. Date
PITT-MT-0006	Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SOW ILM04.0	4	10/06/06
PITT-MT-0008	Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, SOW ILM04.0	2	02/28/00
PITT-MT-0009	Speciated Isotope Dilution Mass Spectrometry, USEPA Method 6800	0	08/04/06
PITT-MT-0020	Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry (ICPMS) for Methods 200.8, 6020 & ILM05.2	2	05/19/05
PITT-MT-0028	Operation of Leeman PS200 (Automated) for Mercury Analysis	0	08/07/98
PITT-MT-I040	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 200.7 CLP-M, SOW ILMO4.0	0	04/15/97
C-OP-0001	Extraction and Cleanup of Organic Compounds from Waters and Solids, Based on SW-846 3500 Series, 3600 Series and 8151A	7	10/20/05
PITT-OP-0011	Extractable Residue (Lipids) from Animal Tissue	1	01/28/03
PITT-OP-0020	Standard Elutriate Test (SET)	1	01/04/06
PITT-OP-0021	Modified and Effluent Elutriate Tests (MET and EET)	0	01/28/03
PITT-OP-0022	Illinois Resuspension Tests	1	10/06/06
PITT-OP-0023	DREDGING ELUTRIATE TEST (DRET)	1	10/06/06
PITT-PM-0001	Project Management	1	9/29/2006
PITT-PM-W-0001	Bottle Kit Guide	0	08/22/06
C-QA-0004	Independent QA Data Review	3	04/01/03
C-QA-010	Non Conformance & Corrective Action System	3	04/01/03
C-QA-013	Employee Orientation & Training	2	04/01/03
PITT-LQM	**Laboratory Quality Manual**	**6**	**01/05/07**
PITT-QA-0002	Statistical Evaluation of Data and Development of Control Charts	1	03/31/03
PITT-QA-0003	Glassware Clean-up for Organic/Inorganic Procedures	1	03/25/02
PITT-QA-0006	Procurement of Standards and Materials; Labeling and Traceability	2	04/01/03
PITT-QA-0007	Determination of Method Detection Limits and Instrument Detection Limits	0	01/05/07

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Table 8.2-2

Standard Operating Procedures

SOP No.	Title	Rev. No.	Rev. Date
	Instrument Detection Limits		
PITT-QA-0008	Temperature Monitoring of Refrigerated Areas and Ovens	1	04/01/03
PITT-QA-0010	Tracking, Review and Revision of SOPs	2	05/24/06
PITT-QA-0012	Daily, Quarterly, Annual Balance Calibration and Class S Weights	1	04/01/03
PITT-QA-0013	Thermometer Calibration and Record Keeping	2	07/15/03
PITT-QA-0017	Aqueous Pipette Calibration – Gravimetric Method	2	04/01/03
PITT-QA-0017a	Container Accuracy Verification – Gravimetric	1	04/01/03
PITT-QA-0019	Records Information Management	1	04/01/03
PITT-QA-0020	Report Production	1	09/01/06
PITT-QA-0022	Equipment Maintenance	1	04/01/03
PITT-QA-0051	Sample Receiving and Chain of Custody	7	10/02/06
PITT-QA-0054	Bottle and Cooler Preparation	0	09/15/06
QA-003	Quality Control Program	3	03/25/02
QA-004	Rounding and Significant Figures	2	04/01/03
QA-008	Data Recording Requirements	3	04/01/03
QA-009	Reporting Limits	4	03/25/02
QA-010	Maintaining Time Integrity	3	04/01/03
QA-012	Technical Data Review Requirements	2	04/01/03
QA-013	Procedures to Address Customer Complaints	2	04/01/03
C-WC-0001	Total Organic Halides in Waters by SW-846 Method 9020B	3	06/21/02
C-WC-0002	Determination of Solids in Waters and Wastes (Methods 160.1/160.2/160.3/160.4 & 2540C/2540D/2540B/2540E)	4	10/12/05
C-WC-0003	HEM / SGT-HEM by Method 1664 (Formerly Oil and Grease / TPH)	1	11/15/99
C-WC-0004	Ignitability of Solids for Waste Characterization EPA SW-846 Chapter 7, Section 7.1	1	10/19/05
PITT-IP-0020	Flash Point by Pensky-Martens Closed Tester	3	10/25/05
PITT-WC-0002	Color, Method 110.2	2	09/01/06
PITT-WC-0003	Alkalinity, EPA Method 310.1 and SM Method 2320B	3	10/12/05

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Table 8.2-2

Standard Operating Procedures

SOP No.	Title	Rev. No.	Rev. Date
PITT-WC-0004	Hardness, Total (mg/L as CaCO3) by Method 130.2/Method 2340C; and Calcium Hardness or Total Calcium by Method 215.2	5	10/12/05
PITT-WC-0005	Turbidity by Method 180.1	2	10/12/05
PITT-WC-0007	Nitrate/Nitrite, Nitrite, EPA Method 353.2	6	10/18/05
PITT-WC-0009	Performance Checks on Spectronic 21 and Model 1001 Spectro-Photometers	1	02/02/99
PITT-WC-0010	Total Sulfide as Acid Soluble Sulfide, Method 9030B	5	10/26/05
PITT-WC-0011	Chloride (Automated), Method 325.2	6	09/08/06
PITT-WC-0012	pH, Specific Conductance, Alkalinity, Hardness, Fluoride, and Acidity (Automatic Titrator)	2	10/12/05
PITT-WC-0014	Nitrogen, Ammonia (Automated), Method 350.1	3	10/18/05
PITT-WC-0015	Chromium, Hexavalent (Colorimetric) by SM3500-Cr-D, SW846 3060A/7196	7	10/26/06
PITT-WC-0016	Biochemical Oxygen Demand (BOD) and Carbonaceous Biochemical Oxygen Demand (CBOD) by Dissolved Oxygen Probe	7	10/13/05
PITT-WC-0017	Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC)	3	10/26/05
PITT-WC-0018	Cyanide – Semi-Automated, Total Cyanide in Water (Method 335.4) and Soil Analyses (Method 9012A)	9	09/08/06
PITT-WC-0020	Percent Moisture and Ash	3	09/01/06
PITT-WC-0022	Fluoride, Method 340.2, SM4500F-C	3	10/13/05
PITT-WC-0026	PH Electrometric by 150.1, 9045C, and 9040B	5	11/10/05
PITT-WC-0029	Chemical Oxygen Demand, Low Level, Method 410.4	1.1	10/25/05
PITT-WC-0032	Cyanide Analysis in Water by CLP	0	12/15/96
PITT-WC-0033	DI-Leachate Procedure for Solids - Soil Extraction for Common Anions	3	09/15/06
PITT-WC-0034	Paint Filter Liquids Test, SW-846 Method 9095A	0	12/07/98
PITT-WC-0037	Sulfate, Turbidimetric, Method 375.4	5	10/18/05
PITT-WC-0038	Phenolics (Automated), Method 420.1/420.2, SW-846 9065/9066	4	09/06/05
PITT-WC-0040	Cyanide Analysis in Soil by CLP	1	04/28/05

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Table 8.2-2

Standard Operating Procedures

SOP No.	Title	Rev. No.	Rev. Date
PITT-WC-0054	Soxhlet Extraction Method for Oil and Grease in Sludge and Sediment by SW-846 Method 9071A	2	04/28/05
PITT-WC-0058	Total Organic Carbon Analysis for Solid Matrices by Walkley Black	2	09/07/06
PITT-WC-0059	Acidity of Water and Waste Water, Method 305.1	1	09/07/06
PITT-WC-0062	Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated Hexane Extractable Material (SGT-HEM; TPH) by Method 1664A	4	11/16/05
PITT-WC-0080	Specific Conductance by 120.1, 2510B, and 9050	1	10/13/05
PITT-WC-0084	Determination of Inorganic Anions by Ion Chromatography EPA Method 300 SW-846 Method 9056	5	09/15/06
PITT-WC-1677	Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA) Method 1677	4	09/08/06
PITT-WC-1991	Acid Volatile Sulfides (AVS) and Simultaneously Extracted Metals (SEM) in Sediment	0	01/28/03

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Table 8.2-3-1

Wet Chemistry Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Acidity	Water	EPA 305.1	---	---
	Waste	EPA 305.1	---	---
	Solid	EPA 305.1 (M)	---	---
Alkalinity	Water	EPA 310.1 2320B	---	---
	Waste	---	---	---
	Solid	310.1 (M)	---	---
Biochemical Oxygen Demand (plus CBOD)	Water	EPA 405.1 5210B	---	---
	Solid	EPA 405.1	---	5210B
Bromide	Water	EPA 300.0	SW 9056	---
	Waste	EPA 300.0	SW 9056	---
	Solid	EPA 300.0 (M)	SW 9056	---
Chemical Oxygen Demand	Water	EPA 410.4	---	---
	Solid	EPA 410.4	---	---
Chloride	Water	EPA 300.0 EPA 325.2	SW 9056	---
	Waste	EPA 300.0	SW 9056	---
	Solid	EPA 300.0 (M)	SW 9056	---
Chromium, Hexavalent	Water	SM 3500-Cr-D	SW 7196A/ 6800	---
	Solid	SM 3500-Cr-D	SW 3060A/7196A/6800	---
	Waste	---	---	---
Color	Water	EPA 110.2	---	---
	Waste	---	---	---
	Solid	---	---	---
Specific Conductance	Water	EPA 120.1	SW 9050A	---
	Solid	EPA 120.1	---	---
	Waste	---	SW 9050A	---
Cyanide (Total)	Water	EPA 335.4	SW 9012A	ILM04.0/ILM04.1
	Waste	---	SW 9012A	ILM04.0/ILM04.1

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Table 8.2-3-1

Wet Chemistry Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
	Solid	---	SW 9012A	ILM04.0/ILM04.1
Cyanide (Available)	Water	EPA 1677	---	---
	Waste	---	---	---
	Solid	---	---	---
Fluoride	Water	EPA 300.0 EPA 340.2	SW 9056	---
	Waste	EPA 340.2 (M) EPA 300.0 (M)	SW 9056	---
	Solid	EPA 340.2 (M) EPA 300.0 (M)	SW 9056	---
Ignitability (Flashpoint)	Waste	---	SW 1010/ 1020	---
	Solid	---	SW 7.1.2	---
Hardness	Water	EPA 130.2 SM2340B	---	---
	Waste	---	---	---
Moisture	Solid	---	SW 160.2 (M)	CLP
Nitrogen, Ammonia	Water	EPA 350.1	---	---
	Waste	EPA 350.1	---	---
	Solid	EPA 350.1	---	---
Nitrite (NO_2)	Water	EPA 300.0 EPA 353.2	SW 9056	---
	Waste	EPA 300.0 (M)	SW 9056	---
	Solid	EPA 300.0(M)	SW 9056	---
Nitrate (NO_3)	Water	EPA 300.0 EPA 353.2	SW 9056	---
	Waste	EPA 300.0 (M)	SW 9056	---
	Solid	EPA 300.0 (M) EPA 353.2 (M)	SW 9056	---
Nitrate plus Nitrite	Water	EPA 353.2	---	---
	Solid	EPA 353.2	---	---
Oil and Grease & NPM	Water	EPA 1664A	SW 9071B	---
	Waste	EPA 1664A	---	---

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Table 8.2-3-1

Wet Chemistry Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
HEM / HEM-SGT	Solid	---	---	---
Ortho-phosphate o-PO_4	Water	EPA 300.0	SW 9056	---
	Waste	EPA 300.0 (M)	SW 9056	---
	Solid	EPA 300.0 (M)	SW 9056	---
Paint Filter Liquids Test	Water	---	---	---
	Waste	---	SW 9095A	---
	Solid	---	SW 9095A	---
pH	Water	EPA 150.1	SW 9040B	---
	Waste	---	SW 9045C	---
	Solid	---	SW 9045C	---
Phenolics	Water	EPA 420.2	SW 9065 SW 9066	---
	Waste	---	SW 9065 SW 9066	---
	Solid	---	SW 9065 SW 9066	---
Sulfate (SO_4)	Water	EPA 300.0 EPA 375.4	SW 9056	---
	Waste	EPA 300.0 (M) EPA 375.4	SW 9056	---
	Solid	EPA 300.0 (M)	SW 9056	---
Sulfide	Water	EPA 376.1	SW 9034	---
	Solid	---	SW 9030B/9034	---
Total Organic and Inorganic Carbon (TOC & TIC)	Water	EPA 415.1	SW 9060	---
	Waste	---	---	---
	Solid	---	Walkley-Black	Lloyd Khan
Total Petroleum Hydro-carbons	Water	EPA 1664 (SGT-HEM) 418.1	---	---
	Waste	EPA 1664 (SGT-HEM)	---	---
	Solid	418.1	---	---

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Table 8.2-3-1

Wet Chemistry Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Total Solids	Water	EPA 160.3	---	---
	Waste	EPA 160.3	---	---
	Solid	EPA 160.3 (M)	---	---
Total Dissolved Solids	Water	EPA 160.1	---	---
Total Suspended Solids	Water	EPA 160.2	---	---
Volatile and Volatile Suspended Solids	Water	EPA 160.4	---	---
Settleable Solids	Water	EPA 160.5	---	---

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Table 8.2-3-2

Methods for Mercury by Cold Vapor Atomic Absorption

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Mercury	Water	EPA 245.1	EPA 7470A	ILM04.0/ILM04.1
	TCLP Leachate	---	EPA 7470A	---
	Waste	---	EPA 7471A	ILM04.0/ILM04.1
	Solid	---	EPA 7471A	ILM04.0/ILM04.1

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Table 8.2-3-3

Methods for Metals by ICP & ICPMS

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Aluminum	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Antimony	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Arsenic	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Barium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Beryllium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Boron	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Calcium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2

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Table 8.2-3-3

Methods for Metals by ICP & ICPMS

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Cadmium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Cobalt	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Chromium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Hexavalent Chromium	Water	---	EPA 6800	---
	Waste	---	---	---
	Solid	---	EPA 6800	---
Copper	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Cobalt	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Iron	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2

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Table 8.2-3-3

Methods for Metals by ICP & ICPMS

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Lead	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Lithium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	---	EPA 6010B	---
Magnesium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Manganese	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Molybdenum	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Nickel	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Potassium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2

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Table 8.2-3-3

Methods for Metals by ICP & ICPMS

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Selenium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Silicon	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	---	EPA 6010B	---
Silver	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Sodium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Strontium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Tin	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Thallium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2

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Table 8.2-3-3

Methods for Metals by ICP & ICPMS

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Titanium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Vanadium	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
Zinc	Water	EPA 200.7/200.8	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Waste	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2
	Solid	---	EPA 6010B/6020	ILM04.0/ILM04.1/ILM05.2

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Table 8.2-3-4

Metals Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water	---	EPA 1311	---
	Waste	---	EPA 1311	---
	Solid	---	EPA 1311	---
ICP Metals	Water	EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate	---	EPA 3010A	---
	Waste	---	EPA 3050B	---
	Solid	EPA 200.7	EPA 3050B	---
CVAA	Water	EPA 245.1	EPA 7470A	---
	TCLP Leachate	---	EPA 7470A	---
	Waste	---	EPA 7471A	---
	Solid	---	EPA 7471A	
ICPMS	Water	200.8	EPA 3020A	---
	TCLP Leachate	---	EPA 3020A	---
	Waste	---	EPA 3050B	---
	Solid	---	EPA 3050B/3060A (Cr VI – EPA 6800)	---

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Table 8.2-3-5

Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	---	EPA 5030B	OLM04.2
	TCLP Leachate	---	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	OLM04.2
	Solid	---	EPA 5035	OLM04.2
Semivolatiles by GC/MS	Water	---	EPA 3510C EPA 3520C	OLM04.2
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3580A	OLM04.2
	Solid	---	EPA 3550B EPA 3580A	OLM04.2
PAHs by GC/MS/SIM (other analytes are available)	Water	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3580A	---
	Solid	---	EPA 3550B EPA 3580A	---
Pesticides/ PCBs by GC	Water	---	EPA 3510C EPA 3520C	OLM04.2
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3580A	OLM04.2
	Solid	---	EPA 3550B	OLM04.2
Pesticides (Organophos-phorus) by GC	Water	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3580A	---
	Solid	---	EPA 3550B	---
PAHs by HPLC	Water	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3580A	---
	Solid	---	EPA 3550B	---

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Table 8.2-3-5

Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Herbicides by GC	Water	---	EPA 8151A	---
	TCLP Leachate	---	EPA 8151A	---
	Waste	---	EPA 8151A	---
	Solid	---	EPA 8151A	---

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Table 8.2-3-5

Organic Methods of Analysis

Analytical Parameters		Fields of Testing		
	Matrix	CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	---	EPA 8260B	OLM04.2/
	TCLP Leachate	---	EPA 8260B	---
	Waste	---	EPA 8260B	OLM04.2
	Solid	---	EPA 8260B	OLM04.2
Semivolatiles by GC/MS	Water	---	EPA 8270C	OLM04.2/
	TCLP Leachate	---	EPA 8270C	---
	Waste	---	EPA 8270C	OLM04.2
	Solid	---	EPA 8270C	OLM04.2
PAHs by GC/MS/SIM (other analytes are available)	Water	---	EPA 8270C SIM	---
	Waste	---	EPA 8270C SIM	---
	Solid	---	EPA 8270C SIM	---
Pesticides/ PCBs by GC	Water	---	Pesticides 8081A PCBs 8082	OLM04.2/
	TCLP Leachate	---	Pesticides 8081A PCBs 8082	---
	Waste	---	Pesticides 8081A PCBs 8082	OLM04.2
	Solid	---	Pesticides 8081A PCBs 8082	OLM04.2
Pesticides (Organophos-phorus) by GC	Water	---	EPA 8141A	---
	Waste	---	EPA 8141A	---
	Solid	---	EPA 8141A	---
PAHs by HPLC	Water	---	EPA 8310	---
	Waste	---	EPA 8310	---
	Solid	---	EPA 8310	---
Phenoxyacid Herbicides by GC	Water	---	EPA 8151A	---
	TCLP Leachate	---	EPA 8151A	---
	Waste	---	EPA 8151A	---

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Table 8.2-3-5

Organic Methods of Analysis

Analytical Parameters		Fields of Testing		
	Matrix	**CWA**	**RCRA (SW846)**	**Other**
	Solid	---	EPA 8151A	---

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Table 8.2-4-1

Method 8260B (Standard Low Level) Volatile Organics by GC/MS

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL 5035 Low Level (µg/Kg)	Soil RL 5035 Mid Level (µg/Kg)
Acetone	67-64-1	20	20	1000
Acetonitrile	75-05-8	100	100	5000
Acrolein	107-02-8	100	100	5000
Acrylonitrile	107-13-1	100	100	5000
Allyl chloride	107-05-1	5.0	5	250
Benzene	71-43-2	5.0	5.0	250
Bromobenzene	108-86-1	5.0	5.0	250
Bromochloromethane	74-97-5	5.0	5.0	250
Bromodichloromethane	75-27-4	5.0	5.0	250
Bromoform	75-25-2	5.0	5.0	250
Bromomethane	74-83-9	10	10	500
2-Butanone (MEK)	78-93-3	20	20	1000
n-Butylbenzene*	104-51-8	5.0	5.0	250
sec-Butylbenzene*	135-98-8	5.0	5.0	250
tert-Butylbenzene*	98-06-6	5.0	5.0	250
Carbon disulfide	75-15-0	5.0	5.0	250
Carbon tetrachloride	56-23-5	5.0	5.0	250
Chlorobenzene	108-90-7	5.0	5.0	250
Chloroprene	126-99-8	5.0	5.0	250
Chlorodibromomethane	124-48-1	5.0	5.0	250
Chloroethane	75-00-3	10	10	500
2-Chloroethyl vinyl ether	110-75-8	10	10	500
Chloroform	67-66-3	5.0	5.0	250
Chloromethane	74-87-3	10	10	500
2-Chlorotoluene*	95-49-8	5.0	5.0	250
4-Chlorotoluene*	106-43-4	5.0	5.0	250
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	10	10	500
1,2-Dibromoethane (EDB)	106-93-4	5.0	5.0	250
Dibromomethane	74-95-3	5.0	5.0	250
1,2-Dichlorobenzene	95-50-1	5.0	5.0	250
1,3-Dichlorobenzene	541-73-1	5.0	5.0	250
1,4-Dichlorobenzene	106-46-7	5.0	5.0	250
Trans-1,4-Dichloro-2-butene	110-57-6	5.0	5.0	250
Dichlorodifluoromethane	75-71-8	10	10	500
1,1-Dichloroethane	75-34-3	5.0	5.0	250

*** Additional compounds commonly performed by STL Pittsburgh.**

This is a Controlled Document. When Printed it becomes Uncontrolled.

Table 8.2-4-1

Method 8260B (Standard Low Level) Volatile Organics by GC/MS

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL 5035 Low Level (µg/Kg)	Soil RL 5035 Mid Level (µg/Kg)
1,2-Dichloroethane	107-06-2	5.0	5.0	250
1,1-Dichloroethene	75-35-4	5.0	5.0	250
cis-1,2-Dichloroethene*	156-59-2	5.0	5.0	250
trans-1,2-Dichloroethene	156-60-5	5.0	5.0	250
1,2-Dichloroethene (total)*	540-59-0	5.0	5.0	250
1,2-Dichloropropane	78-87-5	5.0	5.0	250
1,3-Dichloropropane*	142-28-9	5.0	5.0	250
2,2-Dichloropropane*	594-20-7	5.0	5.0	250
1,1-Dichloropropene*	563-58-6	5.0	5.0	250
cis-1,3-Dichloropropene	10061-01-5	5.0	5.0	250
trans-1,3-Dichloropropene	10061-02-6	5.0	5.0	250
1,4-Dioxane	123-91-1	1000	1000	50000
Ethylbenzene	100-41-4	5.0	5.0	250
Ethyl methacrylate	97-63-2	5.0	5.0	250
Hexachlorobutadiene	87-68-3	5.0	5.0	250
2-Hexanone	591-78-6	20	20	1000
Iodomethane	74-88-4	5.0	5.0	250
Isobutanol	78-83-1	400	400	10000
Isopropylbenzene (Cumene)	98-82-8	5.0	5.0	250
p-Isopropyltoluene*	99-87-6	5.0	5.0	250
Methacrylonitrile	126-98-7	5.0	5.0	250
Methylene Chloride	75-09-2	5.0	5.0	250
Methyl methacrylate	80-62-6	5.0	5.0	250
4-Methyl-2-Pentanone	108-10-1	20	20	1000
Methyl-tert-butyl ether (MTBE)*	1634-04-4	5.0	5.0	250
Naphthalene	91-20-3	5.0	5.0	250
Propionitrile	107-12-0	10	10	500
n-Propylbenzene*	103-65-1	5.0	5.0	250
Styrene	100-42-5	5.0	5.0	250
1,1,1,2-Tetrachloroethane	630-20-6	5.0	5.0	250
1,1,2,2-Tetrachloroethane	79-34-5	5.0	5.0	250
Tetrachloroethene	127-18-4	5.0	5.0	250
Toluene	108-88-3	5.0	5.0	250
1,2,3-Trichlorobenzene	87-61-6	5.0	5.0	250
1,2,4-Trichlorobenzene	120-82-1	5.0	5.0	250

*** Additional compounds commonly performed by STL Pittsburgh.**

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Table 8.2-4-1

Method 8260B (Standard Low Level) Volatile Organics by GC/MS

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL 5035 Low Level (µg/Kg)	Soil RL 5035 Mid Level (µg/Kg)
1,1,1-Trichloroethane	71-55-6	5.0	5.0	250
1,1,2-Trichloroethane	79-00-5	5.0	5.0	250
Trichloroethene	79-01-6	5.0	5.0	250
Trichlorofluoromethane	75-69-4	10.0	10	500
1,2,3-Trichloropropane	96-18-4	5.0	5.0	250
1,2,4-Trimethylbenzene*	95-63-6	5.0	5.0	250
1,3,5-Trimethylbenzene*	108-67-8	5.0	5.0	250
Vinyl Acetate	108-05-4	5.0	10	500
Vinyl Chloride	75-01-4	10	10	500
m- & p-Xylenes	5113-90-0	5.0	5.0	250
o-Xylene	95-47-6	5.0	5.0	250
Xylenes (total) *	1330-20-7	5.0	5.0	250
Methyl Acetate	79-20-9	5.0	5.0	250
Cyclohexane	110-82-7	5.0	5.0	250
Methyl Cyclohexane	108-87-2	5.0	5.0	250
112Trichloro122trifluoroethane	76-13-1	5.0	5.0	250

*** Additional compounds commonly performed by STL Pittsburgh.**

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Table 8.2-4-02

Method 8260B Trace Level Volatile Organics by GC/MS

Reporting Limits (RL)[1]

Analyte	CAS Number	Water RL (µg/L)
Acetone	67-64-1	10.0
Acetonitrile	75-05-8	20.0
Acrolein	107-02-8	20.0
Acrylonitrile	107-13-1	10.0
Allyl chloride	107-05-1	1.0
Benzene	71-43-2	1.0
Bromobenzene	108-86-1	1.0
Bromochloromethane	74-97-5	1.0
Bromodichloromethane	75-27-4	1.0
Bromoform	75-25-2	1.0
Bromomethane	74-83-9	1.0
2-Butanone (MEK)	78-93-3	5.0
n-Butylbenzene*	104-51-8	1.0
sec-Butylbenzene*	135-98-8	1.0
tert-Butylbenzene*	98-06-6	1.0
Carbon disulfide	75-15-0	1.0
Carbon tetrachloride	56-23-5	1.0
Chlorobenzene	108-90-7	1.0
Chloroprene	126-99-8	1.0
Chlorodibromomethane	124-48-1	1.0
Chloroethane	75-00-3	1.0
2-Chloroethyl vinyl ether	110-75-8	2.0
Chloroform	67-66-3	1.0
Chloromethane	74-87-3	1.0
2-Chlorotoluene*	95-49-8	1.0
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	1.0
1,2-Dibromoethane (EDB)	106-93-4	1.0
Dibromomethane	74-95-3	1.0
1,2-Dichlorobenzene	95-50-1	1.0
1,3-Dichlorobenzene	541-73-1	1.0
1,4-Dichlorobenzene	106-46-7	1.0
Trans-1,4-Dichloro-2-butene	110-57-6	1.0
Dichlorodifluoromethane	75-71-8	2.0
1,1-Dichloroethane	75-34-3	1.0
1,2-Dichloroethane	107-06-2	1.0
1,1-Dichloroethene	75-35-4	1.0
cis-1,2-Dichloroethene*	156-59-2	1.0
Trans-1,2-Dichloroethene	156-60-5	1.0

*** Additional compounds commonly performed by STL Pittsburgh.**

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Table 8.2-4-02

Method 8260B Trace Level Volatile Organics by GC/MS

Reporting Limits (RL)[1]

Analyte	CAS Number	Water RL (µg/L)
1,2-Dichloroethene (total)*	540-59-0	1.0
1,2-Dichloropropane	78-87-5	1.0
1,3-Dichloropropane*	142-28-9	1.0
2,2-Dichloropropane*	594-20-7	1.0
1,1-Dichloropropene*	563-58-6	1.0
cis-1,3-Dichloropropene	10061-01-5	1.0
Trans-1,3-Dichloropropene	10061-02-6	1.0
1,4-Dioxane	123-91-1	200
Ethylbenzene	100-41-4	1.0
Ethyl methacrylate	97-63-2	1.0
Hexachlorobutadiene	87-68-3	1.0
2-Hexanone	591-78-6	5.0
Iodomethane	74-88-4	1.0
Isobutanol	78-83-1	40.0
Isopropylbenzene (Cumene)	98-82-8	1.0
p-Isopropyltoluene*	99-87-6	1.0
Methacrylonitrile	126-98-7	1.0
Methylene Chloride	75-09-2	1.0
Methyl methacrylate	80-62-6	1.0
4-Methyl-2-Pentanone	108-10-1	5.0
Methyl-tert-butyl ether (MTBE)*	1634-04-4	1.0
Naphthalene	91-20-3	1.0
Propionitrile	107-12-0	2.0
n-Propylbenzene*	103-65-1	1.0
Styrene	100-42-5	1.0
1,1,1,2-Tetrachloroethane	630-20-6	1.0
1,1,2,2-Tetrachloroethane	79-34-5	1.0
Tetrachloroethene	127-18-4	1.0
Toluene	108-88-3	1.0
1,2,3-Trichlorobenzene	87-61-6	1.0
1,2,4-Trichlorobenzene	120-82-1	1.0
1,1,1-Trichloroethane	71-55-6	1.0
1,1,2-Trichloroethane	79-00-5	1.0
Trichloroethene	79-01-6	1.0
Trichlorofluoromethane	75-69-4	1.0
1,2,3-Trichloropropane	96-18-4	1.0
1,2,4-Trimethylbenzene*	95-63-6	1.0

*** Additional compounds commonly performed by STL Pittsburgh.**

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Table 8.2-4-02

Method 8260B Trace Level Volatile Organics by GC/MS

Reporting Limits (RL)[1]

Analyte	CAS Number	Water RL (µg/L)
1,3,5-Trimethylbenzene*	108-67-8	1.0
Vinyl Acetate	108-05-4	1.0
Vinyl Chloride	75-01-4	1.0
m- & p-Xylenes	5113-90-0	1.0
o-Xylene	95-47-6	1.0
Xylenes (total) *	1330-20-7	1.0

*** Additional compounds commonly performed by STL Pittsburgh.**

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Table 8.2-4-03

Method 8270C Semivolatile Organics by GC/MS

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/kg)	Tissue RL (µg/kg)
Acenaphthene	83-32-9	10	330	400
Acenaphthylene	208-96-8	10	330	400
Acetophenone	98-86-2	10	330	---
2-Acetylaminofluorene	53-96-3	20	660	---
4-Aminobiphenyl	92-67-1	50	1600	---
Aniline	62-53-3	10	330	---
Anthracene	120-12-7	10	330	400
Aramite	140-57-8	50	1600	---
Benzenethiol	108-98-5	100	3300	---
Benzidine	92-87-5	100	3300	---
Benzo(a)anthracene	56-55-3	10	330	400
Benzo(b)fluoranthene	205-99-2	10	330	400
Benzo(k)fluoranthene	207-08-9	10	330	400
Benzoic acid	65-85-0	50	1600	2000
Benzo(g,h,i)perylene	191-24-2	10	330	400
Benzo(a)pyrene	50-32-8	10	330	400
Benzyl alcohol	100-51-6	10	330	400
4-Bromophenyl-phenylether	101-55-3	10	330	400
Butyl benzyl phthalate	85-68-7	10	330	400
Carbazole*	86-74-8	10	330	400
4-Chloroaniline	106-47-8	10	330	2000
bis(2-Chloroethoxy)methane	111-91-1	10	330	400
bis(2-Chloroethyl)ether	111-44-4	10	330	400
bis(2-Chloroisopropyl)ether (2,2'-oxybis(1-Chloropropane)	108-60-1	10	330	400
bis(2-Ethylhexyl)phthalate	117-81-7	10	330	400
Chlorobenzilate	510-15-6	10	330	---
4-Chloro-3-methylphenol	59-50-7	10	330	400
2-Chloronaphthalene	91-58-7	10	330	400
2-Chlorophenol	95-57-8	10	330	400

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*** Additional compounds commonly performed by STL Pittsburgh.**

Comments:

A. N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
B. Hexachlorophene is not amenable to analysis by this method.
C. 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.
D. Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

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Table 8.2-4-03

Method 8270C Semivolatile Organics by GC/MS

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/kg)	Tissue RL (µg/kg)
4-Chlorophenyl phenyl ether	7005-72-3	10	330	400
Chrysene	218-01-9	10	330	400
Diallate	2303-16-4	20	660	---
Dibenzo(a,h)anthracene	53-70-3	10	330	400
Dibenzofuran	132-64-9	10	330	400
Di-n-butylphthalate	84-74-2	10	330	400
1,2-Dichlorobenzene	95-50-1	10	330	400
1,3-Dichlorobenzene	541-73-1	10	330	400
1,4-Dichlorobenzene	106-46-7	10	330	400
3,3'-Dichlorobenzidine	91-94-1	50	1600	2000
2,4-Dichlorophenol	120-83-2	10	330	400
2,6-Dichlorophenol	87-65-0	10	330	---
Diethylphthalate	84-66-2	10	330	400
Dimethoate	60-51-5	20	660	---
4-Dimethylaminoazobenzene	60-11-7	20	660	---
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660	---
3,3'-Dimethylbenzidine	95-53-4	50	1600	---
a,a-Dimethylphenethylamine	122-09-8	50	1600	---
2,4-Dimethylphenol	105-67-9	10	330	400
Dimethyl phthalate	131-11-3	10	330	400
1,3-Dinitrobenzene	99-65-0	10	330	---
4,6-Dinitro-2-methylphenol (4,6-Dinitro-o-cresol)	534-52-1	50	1600	2000
2,4-Dinitrophenol	51-28-5	50	1600	2000
2,4-Dinitrotoluene	121-14-2	10	330	400
2,6-Dinitrotoluene	606-20-2	10	330	400
Dinoseb	88-85-7	20	660	---
Di-n-octylphthalate	117-84-0	10	330	400
Diphenylamine	12-39-4	10	330	---

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*** Additional compounds commonly performed by STL Pittsburgh.**

Comments:

A. N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
B. Hexachlorophene is not amenable to analysis by this method.
C. 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.
D. Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

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Table 8.2-4-03

Method 8270C Semivolatile Organics by GC/MS

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/kg)	Tissue RL (µg/kg)
1,2-Diphenylhydrazine	122-66-7	10	330	400
Disulfoton	298-04-4	50	1600	---
Ethyl methanesulfonate	62-50-0	10	330	---
Famphur	52-85-7	100	3300	---
Fluoranthene	206-44-0	10	330	400
Fluorene	86-73-7	10	330	400
Hexachlorobenzene	118-74-1	10	330	400
Hexachlorobutadiene	87-68-3	10	330	400
Hexachlorocyclopentadiene	77-47-4	50	1600	2000
Hexachloroethane	67-72-1	10	330	400
Hexachloropropene	1888-71-7	100	3300	---
Indeno(1,2,3-cd)pyrene	193-39-5	10	330	400
Isodrin	465-73-6	10	330	---
Isophorone	78-59-1	10	330	400
Isosafrole	120-58-1	20	660	---
Kepone	143-50-0	40	1300	---
Methapyrilene	91-80-5	50	1600	---
3-Methylcholanthrene	56-49-5	50	1600	---
Methyl methanesulfonate	66-27-3	10	330	---
1-Methylnapthalene*	90-12-0	10	330	400
2-Methylnaphthalene	91-57-6	10	330	400
Methyl parathion	298-00-0	10	330	---
2-Methylphenol (o-cresol)	95-48-7	10	330	400
4-Methylphenol (p-cresol) (coelutes w/ 3-Methylphenol)	106-44-5 (108-39-4)	10	330	400
Naphthalene	91-20-3	10	330	400
1,4-Napthoquinone	130-15-4	50	1600	---
1-Naphthylamine	134-32-7	10	330	---
2-Naphthylamine	91-59-8	10	330	---
2-Nitroaniline	88-74-4	50	1600	2000
3-Nitroaniline	99-09-2	50	1600	2000
4-Nitroaniline	100-01-6	50	1600	2000
Nitrobenzene	98-95-3	10	330	400

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*** Additional compounds commonly performed by STL Pittsburgh.**

Comments:

A. N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
B. Hexachlorophene is not amenable to analysis by this method.
C. 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.
D. Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

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Table 8.2-4-03

Method 8270C Semivolatile Organics by GC/MS

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/kg)	Tissue RL (µg/kg)
2-Nitrophenol	88-75-5	10	330	400
4-Nitrophenol	100-02-7	50	1600	2000
4-Nitroquinoline-1-oxide	56-57-5	100	3300	---
N-Nitroso-di-n-butylamine	924-16-3	10	330	---
N-Nitrosodiethylamine	55-18-5	10	330	---
N-Nitrosodimethylamine	62-75-9	10	330	400
N-Nitrosodiphenylamine (Diphenylnitrosamine)	86-30-6	10	330	400
N-Nitroso-di-n-propylamine	621-64-7	10	330	400
N-Nitrosomethylethlyamine	10595-95-6	10	330	---
N-Nitrosomorpholine	59-89-2	10	330	---
N-Nitrosopiperidine	100-75-4	10	330	---
N-Nitrosopyrrolidine	930-55-2	10	330	---
5-Nitro-o-toluidine	99-55-8	20	660	---
Parathion	56-38-2	50	1600	---
Pentachlorobenzene	608-93-5	10	330	---
Pentachloroethane*	76-01-7	50	1600	---
Pentachloronitrobenzene	82-68-8	50	1600	---
Pentachlorophenol	87-86-5	50	1600	2000
Phenacetin	62-44-2	20	660	---
Phenanthrene	85-01-8	10	330	400
Phenol	108-95-2	10	330	400
4-Phenylenediamine	106-50-3	200	6600	---
Phorate	298-02-2	50	1600	---
2-Picoline	109-06-8	20	660	---
Pronamide	23950-58-5	20	660	---
Pyrene	129-00-0	10	330	400
Pyridine	110-86-1	20	660	400
Safrole	94-59-7	20	660	---
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330	---
2,3,4,6-Tetrachlorophenol	58-90-2	10	330	400
2,3,5,6-Tetrachlorophenol*	935-95-5	50	1600	400

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*** Additional compounds commonly performed by STL Pittsburgh.**

Comments:

A. N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
B. Hexachlorophene is not amenable to analysis by this method.
C. 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.
D. Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

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Table 8.2-4-03

Method 8270C Semivolatile Organics by GC/MS

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/kg)	Tissue RL (µg/kg)
Sulfotepp (Tetraethyldithiopyrophosphate)	3689-24-5	50	1600	---
Thionazin	297-97-2	50	1600	---
2-Toluidine	95-53-4	20	660	---
1,2,4-Trichlorobenzene	120-82-1	10	330	400
2,4,5-Tricholorphenol	95-95-4	10	330	400
2,4,6-Trichlorophenol	88-06-2	10	330	400
O,O,O-Triethyl phosphorothioate	126-68-1	50	1600	---
1,3,5-Trinitrobenzene	99-35-4	50	1600	---
6-Methylchrysene	1705-85-7	10	330	---
Dibenz(a,h)acridine	226-36-8	10	330	---
1,4 Dioxane	123-91-1	10	---	---
4,4' Methylenebis(2-chloroaniline)	101-14-4	10	---	---
Benzaldehyde	100-52-7	10	330	---
Caprolactam	105-60-2	10	330	---
1,1'- Biphenyl	92-52-4	10	330	---
Atrazine	912-24-9	10	330	---

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*** Additional compounds commonly performed by STL Pittsburgh.**

Comments:

A. N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
B. Hexachlorophene is not amenable to analysis by this method.
C. 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.
D. Azobenzene is formed by decomposition of 1,2-diphenylhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

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Table 8.2-4-04

Method 8270C Semivolatile Organics by SIM GC/MS

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/kg)	Tissue RL (µg/kg)
Acenaphthene	83-32-9	0.2	6.7	8.0
Acenaphthylene	208-96-8	0.2	6.7	8.0
Anthracene	120-12-7	0.2	6.7	8.0
Benzo(a)anthracene	56-55-3	0.2	6.7	8.0
Benzo(b)fluoranthene	205-99-2	0.2	6.7	8.0
Benzo(k)fluoranthene	207-08-9	0.2	6.7	8.0
Benzo(g,h,i)perylene	191-24-2	0.2	6.7	8.0
Benzo(a)pyrene	50-32-8	0.2	6.7	8.0
bis(2-Chloroethyl)ether	111-44-4	0.2	6.7	---
Carbazole*	86-74-8	0.2	6.7	8.0
2-Chloronaphthalene	91-58-7	0.2	6.7	---
Chrysene	218-01-9	0.2	6.7	8.0
Dibenzo(a,h)anthracene	53-70-3	0.2	6.7	8.0
1,4-Dichlorobenzene	106-46-7	0.2	6.7	8.0
3,3'-Dichlorobenzidine	91-94-1	0.2	6.7	---
2,4-Dichlorophenol	120-83-2	0.2	6.7	---
Fluoranthene	206-44-0	0.2	6.7	8.0
Fluorene	86-73-7	0.2	6.7	8.0
Hexachlorobenzene	118-74-1	0.2	6.7	---
Hexachlorobutadiene	87-68-3	0.2	6.7	---
Indeno(1,2,3-cd)pyrene	193-39-5	0.2	6.7	8.0
2-Methylnaphthalene	91-57-6	0.2	6.7	8.0
1-Methylnaphthalene*	90-12-0	0.2	6.7	8.0
Naphthalene	91-20-3	0.2	6.7	8.0
Nitrobenzene	98-95-3	0.2	6.7	---
N-Nitrosodi-n-propylamine	621-64-7	0.2	6.7	---
2,2'-oxybis(1-Chloropropane)	108-60-1	0.2	6.7	---
Pentachlorophenol	87-86-5	1.0	33	---
Phenanthrene	85-01-8	0.2	6.7	8.0
Phenol	108-95-2	0.2	6.7	---
Pyrene	129-00-0	0.2	6.7	8.0

*** Additional compounds commonly performed by STL Pittsburgh.**

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Table 8.2-4-05

Method 8011 EDB/DBCP

Reporting Limits (RL)

Analyte	CAS Number	Water RL (µg/L)
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	0.02
1,2-Dibromoethane (EDB)	106-93-4	0.02

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Table 8.2-4-06

Method 8081A Water Pesticides (2mL Final Volume) and Method 8082 PCBs

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)
Aldrin	309-00-2	0.01
a-BHC	319-84-6	0.01
b-BHC	319-85-7	0.01
d-BHC	319-86-8	0.01
g-BHC (Lindane)	58-89-9	0.01
Chlordane, Technical	57-74-9	0.10
4,4'-DDD	72-54-8	0.01
4,4'-DDE	72-55-9	0.01
4,4'-DDT	50-29-3	0.01
Dieldrin	60-57-1	0.01
Endosulfan I	959-98-8	0.01
Endosulfan II	33213-65-9	0.01
Endosulfan sulfate	1031-07-8	0.01
Endrin	72-20-8	0.01
Endrin Aldehyde	7421-93-4	0.01
Heptachlor	76-44-8	0.01
Heptachlor Epoxide	1024-57-3	0.01
Aroclor 1016	12674-11-2	0.20
Aroclor 1221	11104-28-2	0.20
Aroclor 1232	11141-16-5	0.20
Aroclor 1242	53469-21-9	0.20
Aroclor 1248	12672-29-6	0.20
Aroclor 1254	11097-69-1	0.20
Aroclor 1260	11096-82-5	0.20
Toxaphene	8001-35-2	0.40

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Table 8.2-4-07

Method 8081A Target Compound List and Appendix IX Pesticides

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/Kg)	Tissue RL (µg/Kg)
Aldrin	309-00-2	0.050	1.7	1.7
a-BHC	319-84-6	0.050	1.7	1.7
b-BHC	319-85-7	0.050	1.7	1.7
d-BHC	319-86-8	0.050	1.7	1.7
g-BHC (Lindane)	58-89-9	0.050	1.7	1.7
Chlordane (Tech.)	57-74-9	0.50	17	17
a-Chlordane	5103-71-9	0.050	1.7	1.7
g-Chlordane	5103-74-2	0.050	1.7	1.7
Chlorobenzilate	510-15-6	0.50	67	---
4,4'-DDD	72-54-8	0.050	1.7	1.7
2,4'-DDD*	53-19-0	0.050	1.7	---
4,4'-DDE	72-55-9	0.050	1.7	1.7
2,4'-DDE*	3424-82-6	0.050	1.7	---
4,4'-DDT	50-29-3	0.050	1.7	1.7
2,4'-DDT*	789-02-6	0.050	1.7	---
Diallate	2303-16-4	1.0	33	---
Dieldrin	60-57-1	0.050	1.7	1.7
Endosulfan I	959-98-8	0.050	1.7	1.7
Endosulfan II	33213-65-9	0.050	1.7	1.7
Endosulfan sulfate	1031-07-8	0.050	1.7	1.7
Endrin	72-20-8	0.050	1.7	1.7
Endrin Aldehyde	7421-93-4	0.050	1.7	1.7
Endrin ketone	53494-70-5	0.050	1.7	1.7
Heptachlor	76-44-8	0.050	1.7	1.7
Heptachlor Epoxide	1024-57-3	0.050	1.7	1.7
Hexachlorobenzene	118-74-1	0.05	1.7	1.7
Isodrin	465-73-6	0.050	17	---
Methoxychlor	72-43-5	0.1	3.3	3.3
Mirex*	2385-85-5	0.05	1.7	1.7
Toxaphene	8001-35-2	2.0	67	67
DCPA (Dacthal) *	1861-32-1	0.1	3.3	3.3
Chlorbenside *	103-17-3	0.1	3.3	3.3

*** Additional compounds commonly perfromed by STL Pittsburgh**

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Table 8.2-4-8

Method 8082 PCBs

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/Kg)	Tissue RL (µg/Kg)
Aroclor 1016	12674-11-2	1.0	33	33
Aroclor 1221	11104-28-2	1.0	33	33
Aroclor 1232	11141-16-5	1.0	33	33
Aroclor 1242	53469-21-9	1.0	33	33
Aroclor 1248	12672-29-6	1.0	33	33
Aroclor 1254	11097-69-1	1.0	33	33
Aroclor 1260	11096-82-5	1.0	33	33

Note: The MDLs provided herein are considered representative, but are subject to change. Please request a list of the current MDLs from the Lab where this is critical for a specific project.

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Table 8.2-4-9

Method 8082 PCBs Congeners[1]

Reporting Limits (RL)[2]

Analyte	CAS Number	Water RL (ng/L)	Soil RL (µg/Kg)	Tissue RL (µg/Kg)
BZ-49	41464-40-8	1.0	0.17	2.0
BZ-115	74472-38-1	1.0	0.17	2.0
BZ-151	52663-63-5	1.0	0.17	2.0
BZ-184	74472-48-3	1.0	0.17	2.0
BZ-126	57465-28-8	1.0	0.17	2.0
BZ-202	2136-99-4	1.0	0.17	2.0
BZ-157	69782-90-7	1.0	0.17	2.0
BZ-167	52663-72-6	1.0	0.17	2.0
BZ-194	35694-08-7	1.0	0.17	2.0
BZ-114	74472-37-0	1.0	0.17	2.0
BZ-123	65510-44-3	1.0	0.17	2.0
BZ-81	70362-50-4	1.0	0.17	2.0
BZ-74	32690-93-0	1.0	0.17	2.0
BZ-189	39635-31-9	1.0	0.17	2.0
BZ-180	35065-29-3	1.0	0.17	2.0
BZ-169	32774-16-6	1.0	0.17	2.0
BZ-170	35065-30-6	1.0	0.17	2.0
BZ-195	52663-78-2	1.0	0.17	2.0
BZ-206	40186-72-9	1.0	0.17	2.0
BZ-209	2051-24-3	1.0	0.17	2.0
BZ-1	2051-60-7	10	1.7	---
BZ-3	2051-62-9	10	1.7	---
BZ-5	16605-91-7	1.0	0.17	---
BZ-8	34883-43-7	1.0	0.17	2.0
BZ-15	2050-68-2	10	1.7	---
BZ-18	37680-65-2	1.0	0.17	2.0
BZ-28	7012-37-5	1.0	0.17	2.0
BZ-31	16606-02-3	1.0	0.17	---
BZ-37	38444-90-5	1.0	0.17	---
BZ-52	35693-99-3	1.0	0.17	2.0
BZ-44	41464-39-5	1.0	0.17	2.0
BZ-66	32598-10-0	1.0	0.17	2.0
BZ-70	32598-11-1	1.0	0.17	---
BZ-90	68194-07-0	1.0	0.17	---
BZ-99	38380-01-7	1.0	0.17	---

[1] **Congener identifications are those proposed by Ballschmiter and Zell in 1980.**

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Table 8.2-4-9

Method 8082 PCBs Congeners[1]

Reporting Limits (RL)[2]

Analyte	CAS Number	Water RL (ng/L)	Soil RL (µg/Kg)	Tissue RL (µg/Kg)
BZ-101	37680-73-2	1.0	0.17	2.0
BZ-87	38380-02-8	1.0	0.17	2.0
BZ-77	32598-13-3	1.0	0.17	2.0
BZ-110	38380-03-9	1.0	0.17	---
BZ-119	56558-17-9	1.0	0.17	---
BZ-118	31508-00-6	1.0	0.17	2.0
BZ-141	52712-04-6	1.0	0.17	---
BZ-149	38380-04-0	1.0	0.17	---
BZ-158	74472-42-7	1.0	0.17	---
BZ-168	59291-65-5	1.0	0.17	---
BZ-177	52663-70-4	1.0	0.17	---
BZ-201	52663-75-9	1.0	0.17	---
BZ-207	52663-79-3	1.0	0.17	---
BZ-153	35065-27-1	1.0	0.17	2.0
BZ-105	32598-14-4	1.0	0.17	2.0
BZ-138	35065-28-2	1.0	0.17	2.0
BZ-187	52663-68-0	1.0	0.17	2.0
BZ-183	52663-69-1	1.0	0.17	2.0
BZ-128	38380-07-3	1.0	0.17	2.0
BZ-156	38380-08-4	1.0	0.17	2.0
BZ-199	52663-73-7	1.0	0.17	2.0
BZ-200	40186-71-8	1.0	0.17	--

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Table 8.2-4-10

Method 8141A Target Compound List and Appendix IX Pesticides

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/Kg)
Azinphos methyl (Guthion)	86-50-0	1.0	33
Bolstar (Sulprofos)	35400-43-2	1.0	33
Chlorpyrifos (Dursban)	2921-88-2	1.0	33
Coumaphos	56-72-4	1.0	33
Demeton-O	298-03-3	1.0	33
Demeton-S	126-75-0	1.0	33
Demeton, (total)	8065-48-3	1.0	33
Diazinon	333-41-5	1.0	33
Dichlorvos (Vapona)	62-73-7	1.0	33
Dimethoate	60-51-5	1.0	33
Disulfoton (Disyston)	298-04-4	1.0	33
EPN	2104-64-5	1.0	33
Ethoprop	13194-48-4	1.0	33
Ethyl parathion	56-38-2	1.0	33
Famphur	52-85-7	1.0	33
Fensulfothion	115-90-2	1.0	33
Fenthion (Baytex)	55-38-9	1.0	33
Malathion	121-75-5	1.0	33
Methyl parathion	298-00-0	1.0	33
Mevinphos (Phosdrin)	7786-34-7	1.0	33
o,o,o-Triethylphosphorothioate	126-68-1	1.0	33
Phorate (Thimet)	298-02-2	1.0	33
Ronnel	299-84-3	1.0	33
Sulfotepp	3689-24-5	1.0	33
Tetrachlorovinphos (Stirophos)	961-11-5	1.0	33
Thionazin	297-97-2	1.0	33
Tokuthion (Prothiophos)	53-10-0	1.0	33
Trichloronate	327-98-0	1.0	33

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Table 8.2-4-11

Method 8151A Herbicides

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/Kg)
2,4-D	94-75-7	4.0	80
2,4-DB	94-82-6	4.0	80
Dalapon	75-99-0	2.0	40
Dicamba	1918-00-9	2.0	40
Dichlorprop	120-36-5	4.0	80
Dinoseb	88-85-7	0.6	12
MCPA	94-74-6	400	8000
MCPP	93-65-2	400	8000
Pentachlorophenol	87-86-5	1.0	10
2,4,5-T	93-76-5	1.0	20
2,4,5-TP (Silvex)	93-72-1	1.0	20

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Table 8.2-4-12

Method 8310 Polynuclear Aromatic Hydrocarbons (HPLC)

Reporting Limits (RL) [1]

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/Kg)	Tissue RL (µg/Kg)
Acenaphthene	83-32-9	1.0	33	33
Acenaphthylene	208-96-8	1.0	33	33
Anthracene	120-12-7	0.20	6.7	6.7
Benzo(a)anthracene	56-55-3	0.20	6.7	6.7
Benzo(a)pyrene	50-32-8	0.20	6.7	6.7
Benzo(b)fluoranthene	205-99-2	0.20	6.7	6.7
Benzo(g,h,i)perylene	191-24-2	0.20	6.7	6.7
Benzo(k)fluoranthene	207-08-9	0.20	6.7	6.7
Carbazole*	86-74-8	1.0	33	33
Chrysene	218-01-9	0.20	6.7	6.7
Dibenzo(a,h)anthracene	53-70-3	0.20	6.7	6.7
Fluoranthene	206-44-0	0.20	6.7	6.7
Fluorene	86-73-7	0.20	6.7	6.7
Indeno(1,2,3-cd)pyrene	193-39-5	0.20	6.7	6.7
1-Methylnaphthalene*	90-12-0	1.0	33	33
2-Methylnaphthalene*	91-57-6	1.0	33	33
Naphthalene	91-20-3	1.0	33	33
Phenanthrene	85-01-8	0.20	6.7	6.7
Pyrene	129-00-0	0.20	6.7	6.7

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Table 8.2-4-13

Method OLM04.2 Volatile Organics by GC/MS

Reporting Limits (RL)

Analyte	CAS Number	Water RL (OLM04.2) (µg/L)	Soil RL (OLM04.2) (µg/Kg)
Acetone	67-64-1	10	10
Benzene	71-43-2	10	10
Bromochloromethane	74-97-5	---	---
Bromodichloromethane	75-27-4	10	10
Bromoform	75-25-2	10	10
2-Butanone (MEK)	78-93-3	10	10
Carbon disulfide	75-15-0	10	10
Carbon tetrachloride	56-23-5	10	10
Chlorobenzene	108-90-7	10	10
Chlorodibromomethane	124-48-1	10	10
Chloroethane	75-00-3	10	10
Chloroform	67-66-3	10	10
Chloromethane	74-87-3	10	10
Cyclohexane	110-82-7	10	10
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	10	10
1,2-Dibromoethane (EDB)	106-93-4	10	10
1,2-Dichlorobenzene	95-50-1	10	10
1,3-Dichlorobenzene	541-73-1	10	10
1,4-Dichlorobenzene	106-46-7	10	10
Dichlorodifluoromethane	75-71-8	10	10
1,1-Dichloroethane	75-34-3	10	10
1,2-Dichloroethane	107-06-2	10	10
1,1-Dichloroethene	75-35-4	10	10
cis-1,2-Dichloroethene	156-59-2	10	10
trans-1,2-Dichloroethene	156-60-5	10	10
1,2-Dichloropropane	78-87-5	10	10
cis-1,3-Dichloropropene	10061-01-5	10	10
trans-1,3-Dichloropropene	10061-02-6	10	10
Ethylbenzene	100-41-4	10	10
2-Hexanone	591-78-6	10	10
Isopropylbenzene (Cumene)	98-82-8	10	10
Methyl acetate	79-20-9	10	10
Methylcyclohexane	108-87-2	10	10
Methylene Chloride	75-09-2	10	10
4-Methyl-2-Pentanone	108-10-1	10	10

MDLs are not utilized in CLP reporting. For GC/MS methods, target hits are reported down to 1/10 of the RL spectra permitting.

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Table 8.2-4-13

Method OLM04.2 Volatile Organics by GC/MS

Reporting Limits (RL)

Analyte	CAS Number	Water RL (OLM04.2) (µg/L)	Soil RL (OLM04.2) (µg/Kg)
Methyl-tert-butyl ether (MTBE)	1634-04-4	10	10
Styrene	100-42-5	10	10
1,1,2,2-Tetrachloroethane	79-34-5	10	10
Tetrachloroethene	127-18-4	10	10
Toluene	108-88-3	10	10
1,2,4-Trichlorobenzene	120-82-1	10	10
1,1,1-Trichloroethane	71-55-6	10	10
1,1,2-Trichloroethane	79-00-5	10	10
Trichloroethene	79-01-6	10	10
Trichlorofluoromethane	75-69-4	10	10
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	10	10
Vinyl Chloride	75-01-4	10	10
Xylenes (total)	1330-20-7	10	10

MDLs are not utilized in CLP reporting. For GC/MS methods, target hits are reported down to 1/10 of the RL spectra permitting.

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Table 8.2-4-14

Method OLM04.2 Semivolatile Organics by GC/MS

Reporting Limits (RL)

Analyte	CAS Number	Water RL (OLM04.2) (µg/L)	Soil RL (OLM04.2) (µg/kg)
Acenaphthene	83-32-9	10	330
Acenaphthylene	208-96-8	10	330
Acetophenone	98-86-2	10	330
Anthracene	120-12-7	10	330
Atrazine	1912-24-9	10	330
Benzaldehyde	100-52-7	10	330
Benzo(a)anthracene	56-55-3	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(g,h,i)perylene	191-24-2	10	330
Benzo(a)pyrene	50-32-8	10	330
1,1'-Biphenyl	92-52-4	10	330
4-Bromophenyl-phenylether	101-55-3	10	330
Butyl benzyl phthalate	85-68-7	10	330
Carbazole	86-74-8	10	330
4-Chloroaniline	106-47-8	10	330
bis(2-Chloroethoxy)methane	111-91-1	10	330
bis(2-Chloroethyl)ether	111-44-4	10	330
bis(2-Ethylhexyl)phthalate	117-81-7	10	330
Caprolactam	105-60-2	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Chlorophenol	95-57-8	10	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Chrysene	218-01-9	10	330
Dibenzo(a,h)anthracene	53-70-3	10	330
Dibenzofuran	132-64-9	10	330
Di-n-butylphthalate	84-74-2	10	330
3,3'-Dichlorobenzidine	91-94-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
Diethylphthalate	84-66-2	10	330
2,4-Dimethylphenol	105-67-9	10	330
Dimethyl phthalate	131-11-3	10	330
4,6-Dinitro-2-methylphenol (4,6-Dinitro-o-cresol)	534-52-1	25	830
2,4-Dinitrophenol	51-28-5	25	830
2,4-Dinitrotoluene	121-14-2	10	330

MDLs are not utilized in CLP reporting. For GC/MS methods, target hits are reported down to 1/10 of the RL spectra permitting.

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Table 8.2-4-14

Method OLM04.2 Semivolatile Organics by GC/MS

Reporting Limits (RL)

Analyte	CAS Number	Water RL (OLM04.2) (µg/L)	Soil RL (OLM04.2) (µg/kg)
2,6-Dinitrotoluene	606-20-2	10	330
Di-n-octylphthalate	117-84-0	10	330
Fluoranthene	206-44-0	10	330
Fluorene	86-73-7	10	330
Hexachlorobenzene	118-74-1	10	330
Hexachlorobutadiene	87-68-3	10	330
Hexachlorocyclopentadiene	77-47-4	10	330
Hexachloroethane	67-72-1	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Isophorone	78-59-1	10	330
2-Methylnaphthalene	91-57-6	10	330
2-Methylphenol (o-cresol)	95-48-7	10	330
4-Methylphenol (p-cresol)	106-44-5	10	330
Naphthalene	91-20-3	10	330
2-Nitroaniline	88-74-4	25	830
3-Nitroaniline	99-09-2	25	830
4-Nitroaniline	100-01-6	25	830
Nitrobenzene	98-95-3	10	330
2-Nitrophenol	88-75-5	10	330
4-Nitrophenol	100-02-7	25	830
N-Nitrosodiphenylamine (Diphenylnitrosamine)	86-30-6	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
2,2'-oxybis(1-Chloropropane)	108-60-1	10	330
Pentachlorophenol	87-86-5	25	830
Phenanthrene	85-01-8	10	330
Phenol	108-95-2	10	330
Pyrene	129-00-0	10	330
2,4,5-Tricholorphenol	95-95-4	25	830
2,4,6-Trichlorophenol	88-06-2	10	330

MDLs are not utilized in CLP reporting. For GC/MS methods, target hits are reported down to 1/10 of the RL spectra permitting.

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Table 8.2-4-15

Method OLM04.2 Pesticides/PCBs by GC

Reporting Limits (RL)

Analyte	CAS Number	Water RL (µg/L)	Soil RL (µg/Kg)
Aldrin	309-00-2	0.050	1.7
a-BHC	319-84-6	0.050	1.7
b-BHC	319-85-7	0.050	1.7
d-BHC	319-86-8	0.050	1.7
g-BHC (Lindane)	58-89-9	0.050	1.7
a-Chlordane	5103-71-9	0.050	1.7
g-Chlordane	5103-74-2	0.050	1.7
4,4'-DDD	72-54-8	0.10	3.3
4,4'-DDE	72-55-9	0.10	3.3
4,4'-DDT	50-29-3	0.10	3.3
Dieldrin	60-57-1	0.10	3.3
Endosulfan I	959-98-8	0.050	1.7
Endosulfan II	33213-65-9	0.10	3.3
Endosulfan sulfate	1031-07-8	0.10	3.3
Endrin	72-20-8	0.10	3.3
Endrin Aldehyde	7421-93-4	0.10	3.3
Endrin ketone	53494-70-5	0.10	3.3
Heptachlor	76-44-8	0.050	1.7
Heptachlor Epoxide	1024-57-3	0.050	1.7
Methoxychlor	72-43-5	0.5	17
Aroclor 1016	12674-11-2	1.0	33
Aroclor 1221	11104-28-2	2.0	67
Aroclor 1232	11141-16-5	1.0	33
Aroclor 1242	53469-21-9	1.0	33
Aroclor 1248	12672-29-6	1.0	33
Aroclor 1254	11097-69-1	1.0	33
Aroclor 1260	11096-82-5	1.0	33
Toxaphene	8001-35-2	5.0	170

MDLs are not utilized in CLP reporting. For GC methods the cut-off for reporting is half the RL.

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Table 8.2-4-16

Mercury by Cold Vapor AA

Reporting Limits (RL) [1]

Element	CAS Number	Water RL (µg/L)	Soil RL (mg/Kg)	Tissue RL (mg/Kg)
Mercury (CLP)	7439-97-6	0.2	0.1	---
(245.1)		0.2	---	---
(7470A/7471A)		0.2	0.033	0.033

Note: The MDLs provided herein are considered representative, but are subject to change. Please request a list of the current MDLs from the Lab where this is critical for a specific project.

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Table 8.2-4-17

Standard ICP Metals Methods 6010B & 200.7

Reporting Limits (RL) [1]

Element	CAS Number	Water 200.7 RL (µgG/L)	Soil 6010B RL[2] (mg/Kg)
Aluminum	7429-90-5	200	20
Antimony	7440-36-0	60	6.0
Arsenic	7440-38-2	300	30
Barium	7440-39-3	200	20
Beryllium	7440-41-7	4.0	0.4
Boron	7440-42-8	200	20
Cadmium	7440-43.9	5.0	0.5
Calcium	7440-70-2	5000	500
Chromium	7440-47-3	10	10
Cobalt	7440-48-4	50	5.0
Copper	7440-50-8	25	2.5
Iron	7439-89-6	100	10
Lead	7439-92-1	100	10
Lithium	7439-93-2	50	5.0
Magnesium	7439-95-4	5000	500
Manganese	7439-96-5	15	1.5
Molybdenum	7439-98-7	40	4.0
Nickel	7440-02-0	40	4.0
Potassium	7440-09-7	5000	500
Selenium	7782-49-2	250	25
Selenium	7782-49-2	250	25
Silica as SiO_2	7404-20-0	1070	107
Silicon	7440-21-3	500	50
Silver	7440-22-4	10	1.0
Sodium	7440-23-5	5000	500
Strontium	7440-24-6	50	5.0
Thallium	7440-28-0	2000	200
Tin	7440-31-5	100	10
Titanium	7440-32-6	50	5.0
Vanadium	7440-62-2	50	5.0
Zinc	7440-66-6	20	2.0

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Table 8.2-4-18

Trace ICP Metals Methods 6010B & 200.7

Reporting Limits (RL) [1]

Element	CAS Number	Water 200.7 RL (µg/L)	Soil 6010B RL[(2)] (mg/Kg)	Tissue 6010B RL (mg/Kg)
Aluminum	7429-90-5	200	20	20
Antimony	7440-36-0	10	1.0	1.0
Arsenic	7440-38-2	10	1.0	1.0
Barium	7440-39-3	200	20	20
Beryllium	7440-41-7	4.0	0.4	0.4
Boron	7440-42-8	200	20	---
Cadmium	7440-43-9	5.0	0.5	0.5
Calcium	7440-70-2	5000	500	500
Chromium	7440-47-3	5.0	0.5	0.5
Cobalt	7440-48-4	50	5.0	5.0
Copper	7440-50-8	25	2.5	2.5
Iron	7439-89-6	100	10	10
Lead	7439-92-1	3.0	0.3	0.3
Magnesium	7439-95-4	5000	500	500
Manganese	7439-96-5	15	1.5	1.5
Molybdenum	7439-98-7	40	4.0	---
Nickel	7440-02-0	40	4.0	4.0
Potassium	7440-09-7	5000	500	500
Selenium	7782-49-2	5.0	0.5	0.5
Silica as SiO_2	7404-20-0	1070	107	---
Silicon	7440-21-3	500	50	---
Silver	7440-22-4	5.0	0.5	0.5
Sodium	7440-23-5	5000	500	500
Strontium	7440-23-5	50	5.0	---
Thallium	7440-42-8	10	1.0	1.0
Tin	7440-31-5	100	10	10
Titanium	7440-32-6	50	5.0	---
Vanadium	7440-62-2	50	5.0	5.0
Zinc	7440-66-6	20	2.0	2.0

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Table 8.2-4-19

ICP Metals Methods ILM04.0/ILM04.1

Reporting Limits (RL)[1] [2]

Element	CAS Number	Water RL (µg/L)	Soil RL (mg/Kg)
Aluminum	7429-90-5	200	40
Antimony	7440-36-0	60	12
Arsenic	7440-38-2	300	60
Barium	7440-39-3	200	40
Beryllium	7440-41-7	5.0	1.0
Boron	7440-42-8	200	40
Cadmium	7440-43-9	5.0	1.0
Calcium	7440-70-2	5000	1000
Chromium	7440-47-3	10	2.0
Cobalt	7440-48-4	50	10
Copper	7440-50-8	25	5.0
Iron	7439-89-6	100	20
Lead	7439-92-1	150	30
Magnesium	7439-95-4	5000	1000
Manganese	7439-96-5	15	3.0
Nickel	7440-02-0	40	8.0
Potassium	7440-09-7	5000	1000
Selenium	7782-49-2	250	50
Silver	7440-22-4	10	2.0
Sodium	7440-23-5	5000	1000
Vanadium	7440-62-2	50	10
Zinc	7440-66-6	20	4.0
Antimony	7440-36-0	10	2.0
Arsenic	7440-38-2	10	2.0
Lead	7439-92-1	3.0	0.60
Selenium	7782-49-2	5.0	1.0
Thallium	7440-28-0	10	2.0

[1] **As per SOW, IDLs are performed quarterly. These are available upon request.**
[2] **Quantitation limits (RL) shown for soils are on a wet weight basis. Quantitation limits on a dry weight basis will be higher. Specific quantitation limits are highly matrix dependent, the quantitation limits listed herein are provided for guidance only, and may not always be achievable.**

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Table 8.2-4-20

Trace ICP Metals Methods 6020 & 200.8

Reporting Limits (RL) [1]

Element	CAS Number	Water 200.8 RL (µg/L)	Water 6020 RL (µg/L	Soil 6020 RL[(2)] (mg/Kg)
Aluminum	7429-90-5	30	30	3
Antimony	7440-36-0	2	2	0.2
Arsenic	7440-38-2	1	1	0.1
Barium	7440-39-3	10	10	1
Beryllium	7440-41-7	1	1	0.1
Boron	7440-42-8	5	5	0.5
Cadmium	7440-43-9	1	1	0.1
Calcium	7440-70-2	100	100	10
Chromium	7440-47-3	2	2	0.2
Cobalt	7440-48-4	0.5	0.5	0.05
Copper	7440-50-8	2	2	0.2
Iron	7439-89-6	50	50	5
Lead	7439-92-1	1	1	0.1
Magnesium	7439-95-4	100	100	10
Manganese	7439-96-5	0.5	0.5	0.05
Molybdenum	7439-98-7	5	5	0.5
Nickel	7440-02-0	1	1	0.1
Potassium	7440-09-7	100	100	10
Selenium	7782-49-2	5.0	5.0	0.5
Silicon	7440-21-3	500	500	50
Silver	7440-22-4	1	1	0.1
Sodium	7440-23-5	100	100	10
Strontium	7440-23-5	5	5	0.5
Thallium	7440-42-8	1	1	0.1
Tin	7440-31-5	5	5	0.5
Titanium	7440-32-6	5	5	0.5
Vanadium	7440-62-2	1	1	0.1
Zinc	7440-66-6	5	5	0.5

Table 8.2-4-21

ICPMS Method 6800 Reporting Limits (RL)

Element	CAS Number	Water 6020 RL (µg/L)	Soil 6020 RL (ug/g)
Hexavalent	7440-47-3	25	2.5

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Chromium			

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Table 8.2-4-22

Wet Chemistry Methods

Reporting Limits (RL)[1]

Parameter	CAS Number	Method	Water RL mg/L	Soil RL mg/Kg
Acidity	76-40-0	EPA 305.1	5.0	---
Alkalinity	477520-60-0	EPA 310.1 SM 2320B	5 5	5.0 ---
Biochemical Oxygen Demand	1-00-2	EPA 405.1 SM 5210B	2.0	120 120
Bromide	28-20-0	EPA 300.0	0.2	---
Chemical Oxygen Demand (COD)	1-00-4	410.4 (Hach)	10	100
Chloride	1-00-3	EPA 300.0 EPA 325.2 SW846 9251	1.0 1.0 1.0	10 10 10
Chromium, hexavalent	18540-29-9	3500-Cr-D SW846 7196A	0.01 0.01	0.1 0.40
Conductivity, specific (Minerals)	1-01-1	EPA 120.1 SW846 9050	1 µmhos/ cm	1 µmhos/ cm
Cyanide	5955-70-0	4500CN-I SW846 9012A EPA 335.1 EPA 335.3 CLP	0.01 0.01 0.01 0.01 0.01	0.5 0.5 --- --- 0.5
Fluoride	66-30-0	EPA 300.0 SM4500F-C EPA 340.2	0.05 0.1 0.1	0.5 1.0 1.0
Hardness	35-50-0	EPA 130.2	5.0	---
Ignitability	1-01-3	SW846 1010	---	---
Nitrogen, Ammonia	310-90-0	EPA 350.1	0.1	5.0
Nitrite as N	15-90-0	EPA 300.0	0.05	0.5
Nitrate plus Nitrite	1-00-5	EPA 353.2	0.1	---
Nitrate as N	25-90-0	EPA 300.0	0.05	0.5
Phenolics, total recoverable	54-30-0	EPA 420.2 SW846 9066	0.01 0.01	0.25 ---

[1] **Quantitation limits (RL) shown for soils are on a wet weight basis. Quantitation limits on a dry weight basis will be higher. Specific quantitation limits are highly matrix dependent, the quantitation limits listed herein are provided for guidance only, and may not always be achievable.**

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Table 8.2-4-22

Wet Chemistry Methods

Reporting Limits (RL)[1]

Parameter	CAS Number	Method	Water RL mg/L	Soil RL mg/Kg
Sulfate	3-03-5	EPA 300.0 EPA 375.4 SW846 9038	1.0 5.0 5.0	--- --- ---
Sulfide	1055-70-0	EPA 376.1 SW846 9030B	1.0 1.0	--- 5.0
Total Organic Carbon (TOC)	1-01-2	Walkley-Black EPA 415.1 SW846 9060 Lloyd Kahn	--- 1.0 1.0 ---	50 --- --- 500
Total Dissolved Solids	1-01-0	EPA 160.1	10	N/A
Total Suspended Solids	1-00-9	EPA 160.2	4.0	N/A
Total Solids	1-00-8	EPA 160.3	10	---
Volatile Suspended Solids	8279840-70-0	EPA 160.4	1	N/A
1664 N-Hexane (HEM)	--	EPA 1664A	5	165
1664 Silica Gel (SGT-HEM)	--	EPA 1664A	5	165
Turbitity (units=NTU)	--	EPA 180.1	1.0 (NTU)	---
Total Inorganic Carbon (TIC)	--	EPA 415.1 SW846 9060	1.0	---
Settleable Solids	3069938-50-0	EPA 160.5 SM 2540F	1mL/L 1mL/L	N/A N/A

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Table 8.4-1

Field Quality Control Samples

Type	Applicability		Accuracy and Precision Application	Introduced By
	Inorganic	Organic		
Trip Blank (volatiles)	No	Yes	Accuracy	Supplier of Containers
Field Blank	Yes	Yes	Accuracy	Field Sampler
Rinsate Blank	Yes	Yes	Accuracy	Field Sampler
Collocated Sample	Yes	Yes	Precision	Field Sampler
Split Sample	Yes	Yes	Precision	Field Sampler
Field Duplicate	Yes	Yes	Precision	Field Sampler
Field Matrix Spike	Yes	Yes	Accuracy	Field Sampler

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Table 8.4-2

Laboratory Quality Control Samples

Type	Frequency	Applicability		Accuracy and Precision Application	Introduced By
		Inorganic	Organic		
Analytical Spike	As specified in methods, or as needed	Yes	No	Accuracy	Analyst/ Prep
Duplicate	1 out of 20 or at least 1/month/run	Yes	Yes	Precision	Analyst/ Prep
Instrument Blank	As specified in methods, or as needed	Yes	Yes	Accuracy	Analyst
Interference Check Sample	As specified in methods	Yes	No	Accuracy	Analyst
Internal Standard	Each sample and standard	No	Yes Method Dependent	Both	Analyst/ Prep
Laboratory Control Sample	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Matrix Spike	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Matrix Spike Duplicate	1 per each group of samples processed up to 20 samples.	Yes	Yes	Both	Analyst/ Prep
Method Blank	1 per each group of samples processed up to 20 samples.	Yes	Yes	Accuracy	Analyst/ Prep
Surrogate	All standards, method blanks, LCS, and samples.	No	Yes Method Dependent	Accuracy	Analyst/ Prep
Yield Monitor	Operation-specific	Yes	No	Accuracy	Prep

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Table 8.4-3

Laboratory Performance Quality Control Samples

Sample/Measurement	Purpose
Method Blanks	Demonstrates that the laboratory systems (*e.g.,* glassware cleaning procedures) and laboratory reagents used for the preparation and analysis of samples have not contributed to a false positive or negative measurement.
Instrument Blank	Demonstrates that the analytical system has not contributed to a false positive or negative measurement.
Laboratory Control Sample	Demonstrates the laboratory's ability to perform an analysis within the performance requirements of the method.

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Table 8.4-4

Matrix Specific Quality Control Samples

Quality Control Sample	Purpose
Duplicate Samples	Estimates the ability of the laboratory to obtain precise measurements on a sample. This measure is dependent on the homogeneity of the sample being duplicated. Solid samples often portray poor sample homogeneity and therefore often have poor duplication with regards to the sample result.
Matrix Spike Sample	Estimates the ability of the laboratory to obtain accurate measurements on a sample. The measure is dependent on the bias a sample matrix may cause regarding a given analyte.
Matrix Spike Duplicate Sample	In addition to verifying the accuracy of the matrix spike sample, the matrix spike duplicate can be used with the matrix spike sample as a measure of precision by calculating the relative percent difference (RPD).

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Table 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[5]	NPDES[1 2 3]		RCRA (SW846) [3 4]	
			Method	Requirements	Method	Requirements
Acidity	Water	100 mL	305.1	250 mL plastic or glass, Cool, 4°C, 14 days	---	Not Applicable
	Solid(5)	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Alkalinity	Water	100 mL	310.1 2320B	250 mL plastic or glass, Cool, 4°C, 14 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Ammonia	Water	400 mL	350.1	500 mL plastic or glass, Cool, 4°C H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Biochemical Oxygen Demand (BOD) and CBOD	Water	200 mL	405.1	1000 mL plastic or glass, Cool, 4°C 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Bromide	Water	100 mL	300.0(7)	250 mL plastic or glass, No preservative required, 28 days	9056	Cool, 4°C, analyze ASAP after collection

[1] Natioinal Pollutant Discharge Elimination System – MCAWW, March 1983
[2] Holding times are calculaed from date of collection
[3] Method not listed in 40 CFR Part 136
[4] Resource Conservation and Recover Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (Augsut 1993), Final Update II (September 1994), Final Update IIB (January 1995) and Final Update III (December 1996)
[5] Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.

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Table 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[5]	NPDES[1 2 3]		RCRA (SW846) [3 4]	
			Method	Requirements	Method	Requirements
	Solid	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
	Waste	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
Chemical Oxygen Demand (COD)	Water	100 mL	410.4	250 mL glass or plastic, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Chloride	Water	50 mL	300.0(7) 325.2	250 mL plastic or glass, No preservative required, 28 days	9056	Cool, 4°C, analyze ASAP after collection.
	Solid	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
	Waste	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
Chromium (Cr^{+6})	Water	100 mL	3500 Cr-D	200 mL quartz, TFE, or polypropylene HNO_3 to pH <2 Cool, 4°C Analyze ASAP after collection	7196A	200 mL plastic or glass, Cool, 4°C, 24 hours
	Solid	Not Applicable	---	Not Applicable	3060A/ 7196A	250 mL plastic or glass, 30 days to digestion, 168 hours after digestion
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Color	Water	100 mL	110.2	250 mL plastic or glass, Cool, 4°C, 48 hours	---	Not Applicable
	Solid	Not	---	Not Applicable	---	Not Applicable

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Table 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[5]	NPDES[1 2 3]		RCRA (SW846) [3 4]	
			Method	Requirements	Method	Requirements
		Applicable				
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Conductivity	Water	100 mL	120.1	200 mL glass or plastic, Cool, 4°C, 28 days	9050A	200 mL glass or plastic, Cool, 4°C, 24 hours
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Cyanide (Total)	Water	IL	335.4	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid(6) Cool, 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid(6) Cool, 4°C, 14 days
	Solid	50g	--	Not Applicable	9012A	8 or 16 oz glass Teflon-lined lids, Cool, 4°C, 14 days
	Waste	50g	--	Not Applicable	9012A	8 or 16 oz glass Teflon-lined lids, Cool, 4°C
Cyanide (Available)	Water	IL	1677	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid(6) Cool, 4°C,	---	Not Applicable
	Solid	50g	---	Not Applicable	---	Not Applicable
	Waste	50g	---	Not Applicable	---	Not Applicable
Flashpoint (Ignitability)	Liquid	Not Applicable	---	Not Applicable	1010/ 1020A	No requirements, 250 mL amber glass, Cool, 4°C

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Table 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[5]	NPDES[1 2 3]		RCRA (SW846) [3 4]	
			Method	Requirements	Method	Requirements
						is recommended
	Solid	Not Applicable	--	Not Applicable	---	Not Applicable
	Waste	Not Applicable	--	Not Applicable	---	Not Applicable
Fluoride	Water	300 mL	300.0(7) 340.2	500 mL plastic, No preservation required, 28 days	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
	Waste	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
Hardness (Total)	Water	50 mL	130.2 2340B	250 mL glass or plastic, HNO_3 to pH < 2, 6 months	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Nitrate	Water	100 mL	300.0(7) 353.2	250 mL plastic or glass, Cool, 4°C, 48 hours.	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
	Waste	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
Nitrite	Water	50 mL	300.0(7) 353.2	250 mL plastic or glass Cool, 4°C, 48 hours	9056	Cool, 4°C, analyze ASAP after collection
	Solid	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
	Waste	Not Applicable	---	Not Applicable	DI Leach/	Not Specified

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Table 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[5]	NPDES[1 2 3]		RCRA (SW846) [3 4]	
			Method	Requirements	Method	Requirements
					9056	
Nitrate-Nitrite	Water	100 mL	353.2	250 mL plastic or glass, H_2SO_4 to pH < 2, 28 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Ortho-phosphate	Water	50 mL	300.0(7)	100 mL plastic or glass, Filter on site Cool, 4°C, 48 hours	9056	Cool, 4°C, analyze ASAP collection
	Solid	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
	Waste	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
Paint Filter Liquids Test	Water	Not Applicable	---	Not Applicable	---	Not Applicable
	Solid	100 g	---	Not Applicable	9095A	Not Specified
	Waste	100 ml or g	---	Not Applicable	9095A	Not Specified
pH (includes Corrosivity)	Water	50 mL	150.1	100 mL plastic or glass. Analyze immediately. This test should be performed in the field.	9040C	100 mL plastic or glass. Analyze immediately. This test should be performed in the field.(8)
	Solid	Not Applicable	---	Not Applicable	9045C	4 oz glass or plastic, Cool, 4°C, Analyze as soon as possible.(8)
	Waste	Not Applicable	---	Not Applicable	9045C	4 oz glass or plastic, Cool, 4°C, Analyze as soon as possible.(8)
Phenolics	Water	100 mL	420.2	500 mL glass, Cool, 4°C,	9065 9066	1 liter glass recommended,

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Table 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[5]	NPDES[1 2 3]		RCRA (SW846) [3 4]	
			Method	Requirements	Method	Requirements
				H_2SO_4 to pH < 2, 28 days		Cool, 4°C, H_2SO_4 to pH < 4, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	9065	Not Specified
Settleable Solids	Water	1000 mL	160.5	1000 mL plastic or glass, Cool, 4°C, 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Specific Conductance	Water	50 mL	120.1	250 mL plastic or glass, Cool, 4°C, 24 hours	9050A	250 mL plastic or glass, Cool, 4°C, 28 days
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Sulfate (SO_4)	Water	100 mL	300.0(7) 375.4	100 mL plastic or glass, Cool, 4°C, 28 days	9056	Cool, 4°C, analyze ASAP collection
	Solid	Not Applicable	---	Not Applicable	DI Leach/ 9056	Not Specified
	Waste	100 mL	---	Not Applicable	DI Leach/ 9056	Not Specified
Sulfide	Water	100 mL	376.1	500 mL plastic or glass, Cool, 4°C, Add 2 mL zinc acetate plus NaOH to pH > 9, 7 days	9030B 9034	500 mL plastic, no headspace, Cool, 4°C, Add 4 drops of 2N zinc acetate per 100 mL of sample, adjust the pH to > 9 with 6 N NaOH solution,

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Table 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[5]	NPDES[1 2 3]		RCRA (SW846) [3 4]	
			Method	Requirements	Method	Requirements
						7 days
	Solid	50 g	---	Not Applicable	9030B 9034	Cool, 4°C, fill surface of solid with 2N Zinc acetate until moistened, store headspace-free
	Waste	50 g	---	Not Applicable	9030B 9034	Cool, 4°C, fill surface of solid with 2N Zinc acetate until moistened, store headspace-free
Total Dissolved Solids (Filterable)	Water	100 mL	160.1	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Total Organic and Inorganic Carbon (TOC & TIC)	Water	100 mL	415.1	100 mL plastic or glass, Cool, 4°C, H_2SO_4 to pH < 2, 28 days	9060	100 mL glass or 40 mL VOA vials, Cool, 4°C, H_2SO_4 or HCl to pH < 2, 28 days
	Solid	Not Applicable	---	Not Applicable	9060	Not Specified
	Waste	Not Applicable	---	Not Applicable	9060	Not Specified
Total Solids	Water	100 mL	160.3	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Total	Water	100 mL	160.2	250 mL plastic or	---	Not Applicable

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Table 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[5]	NPDES[1 2 3]		RCRA (SW846) [3 4]	
			Method	Requirements	Method	Requirements
Suspended Solids (Nonfilterable)				glass, Cool, 4°C, 7 days		
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Turbidity	Water	50 mL	180.1	250 mL plastic or glass, Cool, 4°C, 48 hours	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Volatile Solids	Water	100 mL	160.4	250 mL plastic or glass, Cool, 4°C, 7 days	---	Not Applicable
	Solid	Not Applicable	---	Not Applicable	---	Not Applicable
	Waste	Not Applicable	---	Not Applicable	---	Not Applicable
Water Content	Water	Not Applicable	---	Not Applicable	---	Not Applicable
	Solid	10 g	---	Refer to specific method used	---	Refer to specific method used
	Waste	10 g	---	Refer to specific method used	---	Refer to specific method used
Metals (excludes Hg)	Water	100 mL	200 series	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months	6010B/ 6020	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 6 months
	Solid	200 g	200 series	8 or 16 oz glass or polyethylene container storage at 4 °C	6010B/ 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
	Waste	200 g	200 series	Not Applicable	6010B/ 6020	8 or 16 oz glass or polyethylene

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Table 8.5-1
Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[5]	NPDES[1 2 3]		RCRA (SW846) [3 4]	
			Method	Requirements	Method	Requirements
						container, storage at 4°C, 6 months
Hexavalent Chromium	Water	See SOP PITT-MT-0009	--	--	6800	See SOP PITT-MT-0009
	Solid	See SOP PITT-MT-0009	--	--	6800	See SOP PITT-MT-0009
Mercury (CVAA)	Water	100 mL	245.1	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days	7470A	1 liter glass or polyethylene container, HNO_3 to pH $\leq$ 2, 28 days
	Solid	200 g	245.5	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days	7471A	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days (CORP-MT-0007)
	Waste	200 g	--	Not Applicable	7471A	8 or 16 oz glass or polyethylene container, Cool, 4°C, 28 days (CORP-MT-0007)

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Table 8.5-2

Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[4]	NPDES[1] [2] Method	NPDES[1] [2] Requirements	RCRA (SW846) [2] [3] Method[5]	RCRA (SW846) [2] [3] Requirements
Herbicides	Water	1L-	--	Not Applicable	8151A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	--	Not Applicable	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid, Cool 4 °C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	50 g	--	Not Applicable	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool 4 °C Extraction, 14 days Analysis, 40 days of the start of the extraction
Organo-phosphorus Pesticides	Water	1L	---	Not Applicable	8141A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C,

[1] **National Pollutant Discharge Elimination System – 40 CFR 136, Appendix A**
[2] **Holding times are calculated from the date of collection.**
[3] **Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Wate, Physical/Chemical Methods, Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Updtae III (December 1996)**
[4] **Minimum sample size indicates sample amouint needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquote required.**
[5]

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Table 8.5-2

Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[4]	NPDES[1 2] Method	NPDES[1 2] Requirements	RCRA (SW846) [2 3] Method[5]	RCRA (SW846) [2 3] Requirements
						Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	---	Not Applicable	8141A	4 or 8 oz glass widemouth with Teflon®-lined lid Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	50 g	---	Not Applicable	8141A	4 or 8 oz glass widemouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
Pesticides/ PCBs	Water	1L	---	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8081A 8082	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	---	Not Applicable	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-

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Table 8.5-2

Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[4]	NPDES[1 2] Method	NPDES[1 2] Requirements	RCRA (SW846) [2 3] Method[5]	RCRA (SW846) [2 3] Requirements
						lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	50 g	---	Not Applicable	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C Extraction, 14 days Analysis, 40 days of the start of the extraction
PAHs by HPLC	Water	1L	---	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present. Cool, 4°C, Extraction, 7 days Analysis, 40 days after extraction	8310	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, 40 days of the start of the extraction
	Solid	50 g	---	Not Applicable	8310	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, 40 days of the start of the extraction
	Waste	50 g	---	Not Applicable	8310	4 or 8 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C Extraction, 14 days

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Table 8.5-2

Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[4]	NPDES[1 2] Method	NPDES[1 2] Requirements	RCRA (SW846)[2 3] Method[5]	RCRA (SW846)[2 3] Requirements
						Analysis, 40 days of the start of the extraction
Petroleum Hydrocarbons/Oil and Grease	Water	1 L	1664[(7)]	1 liter glass, Cool, 0-4°C HCl or H_2SO_4 to pH <2 28 days	9070	1 liter glass with Cool, 4°C, HCl to pH <2, 28 days
	Solid	30 g	1664[(7)]	8 or 16 oz. wide mouth glass jar, Cool, 0-4°C, 28 days	9071A	8 oz. glass with Teflon®-lined lid, Holding Time not specified
	Waste	---	---	Not Applicable	9071A	8 oz. glass with Teflon®-lined lid, Holding Time not specified
Semivolatiles by GC/MS	Water	1L	---	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction, 7 days Analysis, 40 days	8270C	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, within 40 days of extraction
	Solid	50 g	---	Not Applicable	8270C	8 or 16 oz glass wide mouth with Teflon-lined lid, Cool, 4°C, Extraction, 14 days Analysis, within 40 days of extraction
	Waste	50 g	---	Not Applicable	8270C	8 or 16 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, within 40 days of extraction

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Table 8.5-2

Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[4]	NPDES[1 2] Method	NPDES[1 2] Requirements	RCRA (SW846) [2 3] Method[5]	RCRA (SW846) [2 3] Requirements
PAHs by GC/MS/SIM (other analytes are available)	Water	1L	---	Not Applicable	8270C SIM	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon, Cool, 4°C, Extraction, 7 days Analysis, within 40 days of extraction
	Solid	50 g	---	Not Applicable	8270C SIM	8 or 16 oz glass wide mouth with Teflon-lined lid, Cool, 4°C, Extraction, 14 days Analysis, within 40 days of extraction
	Waste	50 g	---	Not Applicable	8270C SIM	8 or 16 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction, 14 days Analysis, within 40 days of extraction
Volatile Organics by GC/MS	Water	40 mL	---	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2(8)	8260B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace, Cool, 4°C, Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2(9)
	Solid(5)	5 g or 25 g	--	Not Applicable	8260B	4 or 8 oz glass with Teflon®-lined lid,

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Table 8.5-2

Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[4]	NPDES[1 2] Method	NPDES[1 2] Requirements	RCRA (SW846) [2 3] Method[5]	RCRA (SW846) [2 3] Requirements
						Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCoreTM sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C. (See Note 12 for holding time.)
	Waste	5 g or 25 g	--	Not Applicable	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCoreTM sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore

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Table 8.5-2

Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size[4]	NPDES[1 2] Method	NPDES[1 2] Requirements	RCRA (SW846) [2 3] Method[5]	RCRA (SW846) [2 3] Requirements
						Sampler is 48 hours (before the sample is added to methanol or sodium bisulfate). Cool, 4°C. (See Note 12 for holding time.)

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Table 8.5-3
Sample Containers, Preservatives, and Holding Times for U.S. EPA Contract Laboratory Program Statement of Work

Analytical Parameters	Matrix	Minimum Sample Size	Requirements[1]
Cyanide, Total and Amenable to Chlorination	Water	500 mL	500 mL, glass or polyethylene container, 0.6 g ascorbic acid (only in presence of residual chlorine) NaOH to pH > 12, Cool, 4°C, 12 days
	Soil/Sediment	25 g	8 or 16 oz glass with Teflon-lined lids, Cool, 4°C, 12 days
ICAP and GFAA (excludes mercury)	Water	100 mL	1 liter glass or polyethylene container, HNO_3 to pH =2, 180 days
	Soil/Sediment	25 g	4 or 8 oz glass or polyethylene container, Cool, 4°C, 180 days
Mercury (CVAA)	Water	100 mL	1 liter glass or polyethylene container, HNO_3 to pH =2, 26 days
	Soil/Sediment	25 g	8 or 16 oz glass with Teflon®-lined lids, Cool, 4°C, 26 days
Pesticides/PCBs	Water	1 L	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction within 5 days of sample receipt Analysis within 40 days after start of extraction
	Soil/Sediment	50 g	8 or 16 oz glass wide mouth with Teflon®-lined lid, protect from light, Cool, 4°C, Extraction within 10 days of sample receipt Analysis within 40 days after start of extraction
Semivolatiles	Water	1L	1 liter amber glass with Teflon®-lined lid, Cool, 4°C, Extraction within 5 days of sample receipt Analysis within 40 days after start of extraction
	Soil/Sediment	50 g	8 or 16 oz glass wide mouth with Teflon®-lined lid, Cool, 4°C, Extraction within 10 days of sample receipt Analysis within 40 days after start of extraction

[1] **Holding times are calculated from the verified time of sample receipt.**

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Table 8.5-3
Sample Containers, Preservatives, and Holding Times for U.S. EPA Contract Laboratory Program Statement of Work

Analytical Parameters	Matrix	Minimum Sample Size	Requirements[1]
Volatiles	Water	40 mL	40 mL glass with Teflon®-lined lid, no entrapped air bubbles pH <2, Cool, 4°C, 10 days
	Soil/Sediment	25 g	4 or 8 oz glass with Teflon®-lined lids, Cool, 4°C, 10 days

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Table 8.5-4

Sample Containers, Preservatives, and Holding Times for TCLP[1] and SPLP[2]

Analytical Parameters	Matrix	Minimum Sample Size [3]	TCLP Method 1311 and SPLP Method 1312 Requirements	
			From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Analysis
Mercury	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 28 days	Glass or polyethylene 28 days
Metals (Except mercury)	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 180 days	Glass or polyethylene 180 days
Semivolatiles	Liquid Solid Waste	1L	1L glass, Cool 4°C, 14 days	1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days
Volatiles	Liquid Solid Waste	6 oz	4 oz glass, Cool 4°C, 14 days	40 mL glass, 14 days

[1] **TCLP = Toxicity Characteristic Leaching Procedure**
[2] **SPLP = Synthetic Precipitation Leaching Procedures**
[3] **Smaller sample size is adequate for solid samples or individual fractions. A combination volume of 30 oz. is recommended for semivolatiles and metals. A separate 4 oz. container should always be used for the volatile fraction. Volatile fractions should be stored with minimal headspace.**

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Table 8.5-5

Periodic Equipment Calibrations

Type of Equipment	Calibration Requirements
Balances and Weights	Must be serviced and calibrated annually by an approved vendor. Calibration must be checked daily or before use by analyst with weight(s) classified as Class 1 (formerly termed Class S) by NIST or Class 1 traceable. Acceptance criteria vary according to weight used and accuracy of balance. Acceptance criteria must be documented in the log. All Class 1 weights must be certified by an outside vendor every three years. All non-Class 1 weights must be checked annually against NIST Class 1 weights annually.
Thermometers	Working glass thermometers must be calibrated against a certified NIST thermometer at least annually as described in operation-specific SOPs. Working non-glass thermometers must be calibrated against a certified NIST thermometer annually as described in operation-specific SOPs. The NIST thermometer must be recertified every three years.
Refrigerators/Freezers	Thermometers must be immersed in a liquid such as mineral oil or glycol Temperature of units used for sample or standard storage must be checked daily as described in operation-specific SOPs. Refrigerator acceptance limits: 4°C ± 2°C Freezer acceptance limits: < - 10°C
Ovens	Temperature of units must be checked daily or before use. Acceptance limits vary according to use as described in operation-specific SOPs and must be documented in the temperature log.
Micropipettors	Calibrations are checked gravimetrically as required by the operation-specific SOP. Must be calibrated at the frequency (normally quarterly) required by the manufacturer at a minimum.
Syringes, Volumetric Glassware and Graduated Glassware	All volumetric glassware are purchased as Class A items. Class A items are certified by the manufacturer to be within ± 1% of the measured volume, therefore, calibration of these items by STL® laboratories is not required. Syringes are purchased as specified in programs/methods/client requirements and dispose of every six months. All analysts are trained in the proper use and maintenance of measuring devices to ensure the measurement of standards, reagents and sample volumes are within method tolerances.

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Table 8.6-1
Precision and Accuracy Measurements

Measurement	Definition
Accuracy	The degree of agreement of a measurement with an accepted reference or true value. The only true or known values in the laboratory are spiked samples. Expressed as laboratory control sample (LCS) percent recovery (% R): $LCS\ \%\ Recovery = \frac{X}{t} x\ 100$ where: X = observed concentration t = concentration of spike added Expressed as matrix spike/matrix spike duplicate (MS/MSD) sample percent recovery (% R): $MS / MSD\ \%\ Recovery = \frac{X_s - X}{t} x\ 100$ where: X_s = observed concentration in spiked sample X = observed concentration in unspiked sample t = concentration of spike added
Precision	The measure of analytical reproducibility of two values. Expressed as the relative percent difference (RPD) of two values. $RPD = \left[\frac{\lvert X_1 - X_2 \rvert}{\left(\frac{X_1 + X_2}{2} \right)} \right] x\ 100$ where: X_1 = first observed concentration X_2 = second observed concentration
Arithmetic mean	The average of a set of values. $\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$ where: $\bar{x}$ = the mean x_i = the i^{th} data value n = number of data values
Standard Deviation	A measure of the random (probable) error associated with a single measurement within a data set.

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Table 8.6-1
Precision and Accuracy Measurements

Measurement	Definition
	$s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$ where: s = sample standard deviation $\bar{x}$ = the mean x_i = the i^{th} data value n = number of data values
Quality Control Chart	A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.
ACCURACY	
Upper Control Limit (UCL)	$UCL = \bar{x} + 3s$
Upper Warning Limit (UWL)	$UWL = \bar{x} + 2s$
Lower Warning Limit (LWL)	$LWL = \bar{x} - 2s$
Lower Control Limit (LCL)	$LCL = \bar{x} - 3s$
PRECISION	
RPD	Zero to (mean RPD + 3s)

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Table 8.11-1
Ion Chromatograph[1] Instrument Maintenance Schedule

As Needed	Daily	Weekly	Monthly	Semi-annually
Clean micromembrane suppressor when decreases in sensitivity are observed.	Check plumbing/leaks.	Check pump heads for leaks.	Check all air and liquid lines for discoloration and crimping, if indicated.	Lubricate left hand piston.
Check fuses when power problems occur.		Check filter (inlet)	Check/change bed supports guard and analytical columns, if indicated.	Clean conductivity cell.
Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.	Check pump pressure.			Check conductivity cell for calibration.
De-gas pump head when flow is erratic.	Check conductivity meter.			

[1] **Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.**

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Table 8.11-2

AlpChem Auto Analyzer[1] Instrument Maintenance Schedule

As Needed	Daily	Monthly	Bi-monthly	Annually
Prepare fresh reagents.	Check detector and make sure there are no trapped bubbles in detector cell.	Replace tubing.	Lubricate pump roller.	Clean pump rollers with steel wool and lubricate.
	Check Valves			
	Check Reference source			
Replace pump tubing	Check peristaltic tubing and rollers. Check sampler	Clean pump, diluter, and XYZ Sampler.		
	Clean sample probe shaft.			

[1] **Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.**

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Table 8.11-3	
High Pressure Liquid Chromatograph[1] Instrument Maintenance Schedule	
[2]Daily	**As Needed**
Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent.	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.
Check gas supply.	Oil autosampler slides when sample does not advance.
Flush with an appropriate solvent to remove all bubbles.	Rinse flow cell with 1N nitric acid if sensitivity low.
Pre-filter all samples.	Change pump seals when flow becomes inconsistent.
	Repack front end of column Backflush column.

[1] **Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.**
[2]

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Table 8.11-4

Inductively Coupled Argon Plasma/Mass Spectrometry (ICP/MS)[1] Instrument Maintenance Schedule

Daily	Weekly	Monthly	Quarterly	Annually	As Needed
Check sample waste container level.	Check peristaltic pump: proper roller pressure, sample introduction tubing, correct pump rotation, condition of drain tubing.	Clean all filters and fans.	Replace oil in roughing pumps.	Replace oil in turbo-molecular pump.	Check electronic settings for optimum sensitivity: resolution, mass calibration, ion optics, CEM, deflector voltage.
Check quartz torch condition.	Check condition of sampler and skimmer cones.	Check recirculator water level.			
Measure quartz torch for proper alignment.	Check and drain oil mist eliminator on roughing pumps.				
Clean spray chamber and nebulizer.					
Check oil level of roughing pumps.					

[1] **Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.**

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Table 8.11-5

ICP[1] Instrument Maintenance Schedule

Daily	Monthly or As Needed	Semi-annually	Annually
Check gases Check that argon tank pressure is 50-60 psi and that a spare tank is available. Check aspiration tubing	Clean plasma torch assembly to remove accumulated deposits.	Change vacuum pump oil.	Notify manufacturer service engineer for scheduled preventive maintenance service.
Check vacuum pump gage. (<10 millitorr)	Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance.	Replace coolant water filter. (may require more or less frequently depending on the quality of water)	
Check that cooling water supply system is full and drain bottle is not full. Also that drain tubing is clear, tight fitting and has few bends.	Clean filters on back of power unit to remove dust.		
Check that nebulizer is not clogged.	Replace when needed: peristaltic pump tubing sample capillary tubing autosampler sipper probe		
Check that capillary tubing is clean and in good condition.	Check yttrium position. Check O-rings Clean/lubricate pump rollers.		

[1] **Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.**

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Table 8.11-5

ICP[1] Instrument Maintenance Schedule

Daily	Monthly or As Needed	Semi-annually	Annually
Check that peristaltic pump windings are secure.			
Check that high voltage switch is on.			
Check that exhaust screens are clean.			
Check that torch, glassware, aerosol injector tube, bonnet are clean.			

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Table 8.11-6

Cold Vapor Atomic Absorption (Leeman PS 200)[1] Instrument Maintenance Schedule

Daily	As Needed	Annually
Change drying tube	Change pump tubing	Change Hg lamp.
Check pump tubing/drain tubing	Check/change Hg lamp	
Check gas pressure	Clean optical cell	
Check aperture reading Check tubing	Lubricate pump	

[1] **Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.**

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Table 8.11-7

Gas Chromatograph[1] Instrument Maintenance Schedule

Daily	As Needed	Quarterly/Semi-annually/Annually
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance (e.g. peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.	Quarterly ELCD: change-roughing resin, clean cell assembly. Quarterly FID: clean detector
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.	Semi-annually ECD: perform wipe test.
Check inlets, septa. Replace septum Clean injector port		Annually ELCD: change finishing resin, clean solvent filter. Annually FID: Replace flame tip ECD: detector cleaning and re-foiling, every five years or whenever loss of sensitivity, or erratic response or failing resolution is observed.
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).	
Check reactor temperature of electrolytic conductivity detector.	Replace or repair flow controller if constant gas flow cannot be maintained.	
	Replace fuse.	
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Reactivate external carrier gas dryers.	
Clip column leader	Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. NPD: clean/replace collector assembly. PID: clean lamp window monthly or replace as needed, replace seals. ELCD: check solvent flow weekly, change reaction tube, replace solvent, change	

[1] Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.

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Table 8.11-7

Gas Chromatograph[1] Instrument Maintenance Schedule

Daily	As Needed	Quarterly/Semi-annually/Annually
	reaction gas, clean/replace Teflon® transfer line. ECD: follow manufacturers suggested maintenance schedule	
	Reactivate flow controller filter dryers when presence of moisture is suspected.	
	HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.	
	Purge & trap devices: periodic leak checks quarterly, replace/condition traps (when poor response or disappearance of reactive or poorly trapped compounds), clean sample lines, valves (if they become contaminated), clean glassware. Clean sparger weekly. Check purge flow monthly. Bake trap as needed to correct for high background. Change trap annually, or as needed whenever loss of sensitivity, or erratic response or failing resolution is observed.	
	Purge & trap autosamplers: leak check system, clean sample lines, valves. PTA-30 autosampler also requires cleaning the syringes, frits, valves, and probe needles, adjustment of micro switches, replacement of Teflon® valve, and lubrication of components.	

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Table 8.11-8

Mass Spectrometer[1] Instrument Maintenance Schedule

Daily	Weekly	As Needed [2]	Quarterly	Semi-Annually	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43)	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.	Check ion source and analyzer (clean, replace parts as needed)		Replace the exhaust filters on the mechanical rough pump every 1-2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	Check vacuum, relays, gas pressures and flows	Clean rods	
Check inlets, septa.		Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.	Change oil in the mechanical rough pump. Relubricate the turbomolecular pump-bearing wick.		
Check baseline level.		Repair/replace jet separator.			
Check values		Replace			

[1] **Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.**
[2] **Also see Table 8.11-11 for applicable "As Needed" GC maintenance.**

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Table 8.11-8

Mass Spectrometer[1] Instrument Maintenance Schedule

Daily	Weekly	As Needed [2]	Quarterly	Semi-Annually	Annually
of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		filaments when both filaments burn out or performance indicates need for replacement.			

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Table 8.11-9

Sonicator [1] Instrument Maintenance Schedule

Daily	As Needed
Daily when used: Inspect probe tips for inconsistencies (etching/pitting).	Replace probe tip.
	Disassemble and clean sonicator probe tips.
	Tune sonicator assembly.

Table 8.11-10

Analytical/Top Loading Balances[1] Instrument Maintenance Schedule

Analytical/Top Loading Balances [1]Daily	Annually
Check using Class S-verified weights once daily or before use Clean pan and weighing compartment	Manufacturer cleaning and calibration.

Table 8.11-11

Refrigerators/Walk-In Coolers[1] Instrument Maintenance Schedule

Daily	As Needed
Temperatures checked and logged.	Refrigerant system and electronics serviced.

[1] **Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.**

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Table 8.11-12

Ovens[1] Instrument Maintenance Schedule

Daily	As Needed
Temperatures checked and logged.	Electronics serviced.

Table 8.11-13

Specific Digital Ion Analyzer[1] Instrument Maintenance Schedule

Daily	As Needed
Daily when used: Calibrate with check standards. Inspect electrode daily, clean as needed. Inspect electrode proper levels of filling solutions daily, fill as needed. Clean probe, each use.	Electronics serviced.

Table 8.11-14

Turbidimeter[1] Instrument Maintenance Schedule

Turbidimeter Daily	Monthly	As Needed
Daily when used: Adjust linearity on varying levels of NTU standards. Standardize with NTU standards. Inspect cells.	Clean instrument housing	Electronics serviced.

[1] **Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.**

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Table 8.11-15

Dissolved Oxygen Meter [1] Instrument Maintenance Schedule

Daily	As Needed
Daily when used: Calibrate with check standards. Check probe membrane for deterioration Clean and replace membrane with electrode solution.	Electronics serviced.

Table 8.11-16

Conductance Meter[1] Instrument Maintenance Schedule

Daily	As Needed
Daily when used: Check probe and cables. Standardize with KCl. Inspect conductivity cell	Electronics serviced.

Table 8.11-17

Chemical Oxygen Demand (COD) Reactor[1] Instrument Maintenance Schedule

Daily	As Needed
Daily when used: Calibrate with check standards.	Electronics serviced.

[1] **Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.**

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Table 8.11-18

Spectrophotometer[1] Instrument Maintenance Schedule

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens.	Check the zero %A adjustment.	Clean windows	Check instrument manual.
	Clean sample compartment		Perform wavelength calibration.
	Clean cuvettes		Replace lamp annually or when erratic response is observed.
			Clean and align optics.

Table 8.11-19

pH Meter[1] Instrument Maintenance Schedule

pH Meter As Needed	Daily
Clean electrode.	Inspect electrode. Verify electrodes are properly connected and filled.
Refill reference electrode.	Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer (pH 4.0).

[1] **Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations.**

Table 8.11-20

Alpkem FS3000

1677 Available Cyanide Needed	Daily	Monthly	Bi-monthly
Prepare fresh reagents.	Clean detector cell and make sure there are no trapped bubbles in lines.	Replace tubing.	Lubricate pump roller Replace Diffusion Membrane
Replace pump tubing	Check peristaltic tubing and rollers.		Clean Reference Electrode
			Replace Reference solution

Table 8.11-20A

Konelab

Daily	Weekly	Monthly
Run “Start Up”	Empty liquid waste	Restore adjustments from disk
Review water check	Clean wash wells and tubing to waste	Save database to CD
Empty waste bin	Check for chemical residue	Print – then delete messages
Fill diluent with fresh DI water	Clean off any chemical residue	Print – Water Check
Check waste container	Check syringe plunger Teflon tip	Run Dichromate test at 480nm
Run “Stand By”	Run Dichromate test at 480 nm	Clean and Lube incubator rod
Print or save results to file	Reboot computer	Clean and Lube fetcher rod
Clear daily files		
Clean incubator		

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Table 8.11-21

Total Organic Carbon Analyzer (OI 7000) Instrument Maintenance Schedule

Daily	As Needed	Weekly	Monthly	Semi-Annually
Check: Oxygen supply Persulfate supply Acid supply Carrier gas flow rate (~ 150 cc/min) IR millivolts for stability (after 30 min. warm-up) Reagent reservoirs	Check injection port septum after 50-200 runs. Tube end-fitting connections after 100 hours or use. Indicating drying tube. NDIR zero, after 100 hours of use. Sample pump, after 2000 hours for use. Digestion vessel/condensation chamber, after 2000 hours of use. Permeation tube, after 2000 hours of use. NDIR cell, after 2000 hours of use.	Check liquid-flow-rate-pump-tubing conditions on autosampler Check injection port septum	Clean digestion vessel Clean condenser column Do the leak test	Change pump tubing

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Table 8.11-22

Digestion Block Instrument Maintenance Schedule

Annually
Check temperature with NIST thermometer

Table 8.11-23

Flash Point Tester Instrument Maintenance Schedule

Daily	As Needed
Check tubing. Clean sample cup each use.	Check thermometer against NIST thermometer, when used.
Check gas.	
Clean flash assembly	
Check stirrer	

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Table 9.4-1

Proficiency Testing Programs

PT Sample Program Description	Analysis Performed	Frequency of Participation
Water Pollution Program Samples provided by Environmental Resource Associates, a NIST-approved PT Provider	Trace Metals, Minerals, Nutrients, Demand, PCBs in Water, PCBs in Transformer Oil, Pesticides (Insecticides), Volatile Halocarbons, Volatile Aromatics, Semivolatiles (base/neutrals/acids), Polynuclear Aromatic Hydrocarbons (HPLC) and Miscellaneous (TSS, Cyanide, Total Phenolics)	Semi-annual
Water Supply Program Samples provided by Environmental Resource Associates, a NIST-approved PT Provider	All methods performed for: EPA 1677 low level cyanide	Semi-annual
Hazardous Waste Program Samples provided by Environmental Resource Associates, a NIST-approved PT Provider	Semivolatile Organics (BNA), Pesticides, Herbicides, Volatile Organics, Metals, Anions, PAHs, TPH Gas and Diesel, Explosives	Semi-annual
STL Corporate Double Blind	Volatile Organics, Metals, General Chemistry, Base/Neutral Acid Extractables, Project Management	Annually
Allied Signal	Volatile Organics, Semivolatile Organics, Metals, BOD, COD, TSS, TPH	Annually
STL Pittsburgh Lab Internal PTs	As needed	As a follow-up to unacceptable PTs from other programs.

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Acronyms and Initialisms

A2LA	American Association for Laboratory Accreditation
AA	Atomic Absorption
ANSI	American National Standards Institute
AR/COC	Analysis Request/Chain-of-Custody
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
BFB	Bromofluorobenzene
BLK	Blank
BOD	Biochemical Oxygen Demand
CCC	Calibration Check Compound
CEO	Chief Executive Officer
CF	Calibration Factor
CFR	Code of Federal Regulations
CHP	Chemical Hygiene Plan
CLP	Contract Laboratory Program
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act (Superfund)
COC	Chain-of-Custody
COD	Chemical Oxygen Demand
CRDL	Contract Required Detection Limit
CRQL	Contract Required Quantitation Limit
CSM	Customer Service Manager
CSRM	Certified Standard Reference Material
CST	Customer Service Team
CUR	Condition Upon Receipt
CV	Coefficient of Variation
CVAA	Cold Vapor Atomic Absorption (Spectroscopy)
DFTPP	Decafluorotriphenylphosphine
DOC	Dissolved Organic Carbon
DOE	Department of Energy
DOT	Department of Transportation
DQO	Data Quality Objective
EH&S	Environmental Health and Safety
EPA	(U. S.) Environmental Protection Agency

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Acronyms and Initialisms

FAS	Field Analytical Services
FLAA	Flame Atomic Absorption (Spectroscopy)
FTIR	Fourier Transform Infrared (Spectrometry)
GC	Gas Chromatograph(y)
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption (Spectroscopy)
HDPE	High Density Polyethylene
HPLC	High Performance Liquid Chromatography
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry
ICAP	Inductively Coupled Argon Plasma (Spectroscopy)
ICAP/MS	Inductively Coupled Argon Plasma/Mass Spectrometry
ICS	Interference Check Sample
IDL	Instrument Detection Limit
IR	Infrared (Spectroscopy)
IS	Information Systems
IS	Internal Standard
ISO	International Organization for Standardization
IT	Information Technology
KRI	Key Result Indicator
LAN	Local Area Network
LCL	Lower Control Limit
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LQM	Laboratory Quality Manual
LRGC	Low Resolution Gas Chromatography
LRMS	Low Resolution Mass Spectrometry
LWL	Lower Warning Limit
MBAS	Methylene Blue Active Substance
MDC	Minimum Detectable Concentration
MDL	Method Detection Limit
MS	Matrix Spike

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Acronyms and Initialisms

MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheet
NELAC	National Environmental Laboratory Accreditation Conference
NELAP	National Environmental Laboratory Accreditation Program
NCM	Nonconformance Memo
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards Technology
NMOC	Non-Methane Organic Compounds
NPDES	National Pollutant Discharge Elimination System
NRC	Nuclear Regulatory Commission
NRM	National Reference Material
PAH	Polynuclear Aromatic Hydrocarbons (or PNA)
PC	Personal Computer
PCB	Polychlorinated Biphenyls
PDS	Post Digestion Spike
PE	Performance Evaluation
PEM	Performance Evaluation Mixture
PM	Project Manager
PQL	Practical Quantitation Limit
PSRL	Project-Specific Reporting Limit
PUF	Polyurethane Foam
QA	Quality Assurance
QAPP	Quality Assurance Project Plan or Quality Assurance Program Plan
QAS	Quality Assurance Summary
QC	Quality Control
QS	Quality System
QuantIMS	STL Pittsburgh Laboratory Information Management System
QRI	Quality-Related Item
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RFP	Request for Proposal
RFQ	Request for Quote

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Acronyms and Initialisms

RL	Reporting Limit
RPD	Relative Percent Difference
RRF	Relative Response Factor
RSD	Relative Standard Deviation
RSO	Radiation Safety Officer
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SOW	Statement of Work
SPCC	System Performance Check Compounds
SPLP	Synthetic Precipitation Leaching Procedure
SRL	Standard Reporting Limit
SRM	Standard Reference Material
TCLP	Toxicity Characteristic Leaching Procedure
TIC	Tentatively Identified Compound
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
UCL	Upper Control Limit
UPS	Uninterruptible Power Supply
USEPA	United States Environmental Protection Agency
UWL	Upper Warning Limit
VOA	Volatile Organic Analysis
VOST	Volatile Organic Sampling Train
WAN	Wide Area Network
WS	Water Supply
WP	Water Pollution

Glossary

acceptance limits

Data quality limits specified for analytical method performance.

Accuracy

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. Systematic errors affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100).

Aliquot, aliquant

A measured portion of a sample taken for analysis.

Analytical spike

A sample created by spiking target analytes into a prepared portion of a sample just prior to analysis. (Also see matrix spike.)

Anomaly

See nonconformance.

Areas needing improvement

Represent isolated instances of noncompliance or issues that are judged to have a less immediate impact on data quality. Laboratory management must correct the situation or otherwise ensure that the condition does not recur. This term replaces the previous term used “Observations.”

Arithmetic mean

The arithmetic mean ($\bar{x}$) is the average of a set of values. It is equal to the sum of the observed values divided by the number of observations. Also called "average".

where: $\bar{x}$ = the mean

x_i = the i^{th} data value

$$\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

n = number of data values

Assessment

The evaluation process used to measure the performance or effectiveness of a system and its elements. Assessment is used as an all-inclusive term to denote any of the following: performance, systems, data and compliance audits, management systems reviews, peer reviews, inspections, or spot assessments.

Associate

Employee.

Audit

A planned and documented investigative evaluation of an item or process to determine its adequacy and effectiveness as well as compliance with established procedures, instructions, drawings, quality management plans, and other applicable documents.

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Glossary

Benchmarking A step-by-step method of improving performance by identifying and studying best practices and comparing them to industry practices.
Bias A systematic (consistent) error in test results. Bias is expressed as the difference between the population mean and the true or reference value, or as estimated from sample statistics, the difference between the sample average and the reference value.
Blind performance evaluation sample A sample either submitted to the laboratory or prepared in the laboratory whereby the concentrations of parameters of concern are known by the preparer and not by the laboratory.
Calibration Establishment of a relationship between various calibration standards and the measurements of them obtained by a measurement system, or portions thereof. The levels of the calibration standard should bracket the range of levels at which actual measurements are to be made. Calibration is also the act of making a scheduled comparison of instrument performance against national standards for instruments which measure physical parameters such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.
Calibration curve The graphical relationship between the known values for a series of calibration standards and instrument responses.
Calibration factor (CF) The ratio of the instrument response of an analyte to the amount injected. CFs are used in external standard calibrations. $$CF = \frac{Total\ Area\ of\ Peak}{Mass\ Injected}$$
calibration standard A standard used to quantitate the relationship between the output of a sensor and a property to be measured. Calibration standards should be traceable to standard reference materials (provided by NIST, or other recognized standards agencies) or a primary standard.
Certificate of Analysis A STL report format containing analytical results without supporting/backup information.
certified reference material A reference material accompanied by a certificate issued by an organization certifying the contents and concentration(s) of the material. (See also standard reference material.)
chain-of-custody (COC) A system of documentation demonstrating the physical custody and traceability of samples.
check standard analyses A standard (often a midpoint standard) analyzed at a frequency specified in the method or in an SOP to verify the continuing calibration of the standard curve.

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client

Any individual or organization for whom items or services are furnished or work is performed in response to defined requirements and expectations.

client sample

The material or collection media submitted to the laboratory for analysis. Field QC samples are considered client samples but laboratory QC samples are not counted as client samples when counting samples for QC batches.

Coefficient of variation (relative standard deviation)

A measure of precision (relative dispersion). It is equal to the standard deviation (*s*) divided by the mean ($\bar{x}$) and multiplied by 100 to give a percentage value.

$$CV\ (RSD) = \left(\frac{s}{\bar{x}}\right) x\ 100$$

collocated samples

Independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. The results will indicate sampling as well as analytical variability.

Comparability

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (i.e., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

Completeness

$$\%\ Completeness = \frac{V}{n}\ x\ 100$$

where: V = number of measurements judged valid

n = total number of measurements

Completeness is a measure of the percentage of measurements that are judged to be valid measurements. At a minimum, the objective for completeness of data is 90% for each constituent analyzed. It is usually expressed as a percentage.

Composite

A sample composed of two or more increments.

Control chart

A graphical representation of analytical accuracy. Displays the arithmetic mean of a data set, the upper and lower warning limits and the upper and lower control limits.

Control table

A tabular presentation of test results with respect to time or sequence of measurement, together with limits within which the results are expected to lie when the analytical process is in a state of control.

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Controlled document A document for which the distribution is known. Updates of the document are sent to the original recipients, unless the copy distributed is an uncontrolled copy.
Corrective action A measure taken to rectify conditions adverse to quality and, where necessary, to preclude their recurrence.
Correlation coefficient The correlation coefficient (r) is a determination of how closely data "fits" a straight line. It is a number between -1 and 1 that indicates the degree of linear relationship between two sets of numbers. A correlation coefficient of +1 (usually calculated to three decimal places or 1.000) means the data falls exactly on a straight line with positive slope. A correlation coefficient of -1 (or -1.000) means the data falls exactly on a straight line with negative slope.
Data quality objective (DQO) Data quality objectives (DQOs) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application (EPA 1994). Typically, DQOs are identified during project scope and development of sampling and analysis plans. In this QA manual, however, we refer to only the analytical DQOs because laboratories generally do not have any authority over sample collection, shipment, or other field-related activities that may affect the data quality of the environmental sample before the sample is received in the laboratory. EPA has established six primary analytical DQOs for environmental studies: precision, accuracy, representativeness, completeness, comparability, and detectability. The components of analytical variability (uncertainty) can be estimated when QA and QC samples of the right types and quantities are incorporated into measurement procedures at the analytical laboratory. STL incorporates numerous QA and QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QA and QC samples and their applications, described in Section 8.4 and are selected on the basis of method- or client-specific requirements. Field blanks, field duplicates, and performance evaluation (PE) samples are received from the client as unknown samples. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses may include calibration or instrument blanks, method blanks, background, duplicates, replicates, laboratory control samples (LCSs), calibration standards, matrix spikes (MSs), matrix spike duplicates (MSDs), surrogate spikes, and yield tracers.
data validation See validation - data.
data verification See verification - data.
deficiency See nonconformance or finding.
degrees of freedom The number of independent deviations used in calculating an estimate of the standard deviation. Double blind performance evaluation sample
A sample that contains select parameters at defined levels. The levels are unknown to the laboratory. The laboratory is also unaware that the sample is a performance evaluation sample. Duplicate sample analyses Different aliquots of the same sample are analyzed to evaluate the precision of an analysis.

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Error The difference between an observed or measured value and its true value.
Field blank A blank that is prepared and handled in the field and analyzed in the same manner as its corresponding client samples.
Field matrix spike A sample created by spiking target analytes into a sample in the field at the point of sample acquisition.
Finding Noncompliant practices or policies which have significant adverse impact on data quality, technical defensibility, or regulatory acceptance of data. Findings require immediate attention by the laboratory management and must be resolved to comply with STL's quality documents and laboratory-established procedures often called deficiencies by auditors.
Geometric mean The n^{th} root of the product of all values in a set of n values or the antilogarithm of the arithmetic mean of the logarithms of all the values of a set of n values. The geometric mean is generally used when the logarithms of a set of values are nearly normally (Gaussian) distributed, such as is the case of much population data.
Initial calibration Analysis of a series of analytical standards at different specified concentrations; used to define the linearity and dynamic range of the response of an instrument to the target compounds prior to the analysis of samples.
Inspection Examination or measurement of an item or activity to verify conformance to specific requirements.
Instrument detection limit (IDL) IDL is a calculated estimate of instrument detectability defined by the USEPA Contract Laboratory Program (CLP).
Internal standard (IS) A compound added to every standard, QC sample, client sample, or sample extract at a known concentration prior to analysis for the purpose of quantitation. For example, internal standards are used as the basis for quantitation of the target compounds by GC/MS.
Linear regression A statistical method for finding a straight line that best fits a set of two or more data points, thus providing a relationship between two or more variables.
Laboratory Quality Manual (LQM) The Laboratory Quality Manual (LQM) is a formal document that describes quality systems in terms of organizational structure, functional responsibilities of management, and staff, and lines of authority. The LQM documents the QMS and describes both the organizational and project-specific principles, goals, controls, and tools of the QMS. The LQM provides the criteria and specifications for the generation of environmental analytical data.
Matrix The component or substrate which contains the analyte(s) of interest. Examples of matrices are water, soil or

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sediment, and air. Matrix is not synonymous with phase (liquid or solid).

Matrix effect

An interference in the measurement of analyte(s) in a sample that is caused by materials in the sample. Matrix effects may cause elevated reporting limits or may prevent the acquisition of acceptable results.

Matrix spike (MS)

An aliquot of a matrix fortified (spiked) with known quantities of specific compounds and subjected to an entire analytical procedure in order to indicate the appropriateness of the method for a particular matrix. The percent recovery for the respective compound(s) is then calculated.

Matrix spike duplicate (MSD)

A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

May

Denotes permission but not a requirement.

Mean

See arithmetic mean.

Measurement

The process or operation of ascertaining the extent, degree, quantity, dimensions, or capability with respect to a standard.

Median

The middle value of a set of data when the data set is ranked in increasing or decreasing order.

Method

An assemblage of techniques.

Method blank (MB)

An analytical control consisting of all reagents, which may include internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination. Examples of method blanks are a volume of deionized or distilled laboratory water for water samples, a purified solid matrix for soil/sediment samples, or a generated zero air.

Method detection limit (MDL)

The minimum concentration of an analyte that, in a given matrix and with a specific method, can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is operationally defined as:

$$MDL = s t_{(n-1, \alpha = 0.99)}$$

where:

s = the standard deviation of a number of measurements of a blind or sample matrix containing the analyte at a concentration near the lowest standard recommended in the method and

$t_{(n-1, \alpha = 0.99)}$ = the student's value for a one-sided t-statistic appropriate for the number of samples used to determine (s), at the 99% confidence level and n-1 degrees of freedom.

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Modified method

A standard or reference method which has been changed to meet project or matrix requirements.

Must

Denotes a requirement is mandatory and has to be met.

Notable practices

Laboratory practices that increase effectiveness and quality and represent improvements with respect to conventional laboratory operations.

Nonconformance

An unplanned deviation from an established protocol or plan. The deviation may be the result of STL's actions, then termed a deficiency. If the deviation is the result of events beyond the control of STL, it is termed an anomaly.

Operational calibration

Routinely performed as part of instrument usage, such as the development of a standard calibration curve. Operational calibration is generally performed for instrument systems.

Outlier

A result excluded from the statistical calculations due to being deemed "suspicious" when applying the "Grubbs Test" (or equivalent).

Parameter

A constant or coefficient that describes some characteristic of a population (e.g., standard deviation, mean, regression coefficients). Also, a chemical being measured, i.e., an analyte.

Percent difference

When two independent measurements of the same characteristics are available, it is possible to use the percent difference instead of the coefficient of variation to measure precision.

$$\%D = \left| \frac{X_1 - X_2}{X_1} \right| x\ 100\%$$

where: $\%D$ = percent difference

X_1 = first value

X_2 = second value

Percent recovery

A measure of accuracy determined from the comparison of a reported spike value to its true spike concentration.

$$\%R = \frac{observed\ conc. - sample\ conc.}{true\ spike\ conc.} x\ 100\%$$

Performance audit

See performance evaluation.

Performance evaluation (PE)

A type of audit in which a known or characterized value is compared to the result obtained through the routine

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analysis of the sample in the laboratory to evaluate the proficiency of an analyst or laboratory.
Periodic calibration A calibration that is performed at prescribed intervals for equipment such as balances, thermometers, and balance weights. In general, they are performed on equipment that are distinct, singular purpose units, and are relatively stable in performance.
Population A generic term denoting any finite or infinite collection of individual things, objects, or events.
practical quantitation limit (PQL) The lowest concentration a method can reliably achieve within limits of precision and accuracy and is derived from empirical, matrix-free method performance studies.
Precision Precision is an estimate of variability, that is, it is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. The precision of a measurement system is affected by random errors. Precision is expressed either as relative standard deviation (RSD) for replicate measurements greater than two or as relative percent difference (RPD) for duplicate measurements. Table 8.6-1 illustrates the formulae used to calculate units of precision (i.e., RSD and RPD).
Preventive maintenance An organized program within STL laboratories of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use.
Primary standard A material having a known, stable property that can be accurately measured or derived from established physical or chemical constants. It is readily reproducible and can be accepted (within stated limits) and used to establish the same value of another substance or item.
Procedure Detailed instructions to permit replication of a method. (See standard operating procedure.)
Proficiency testing A series of planned tests which will determine the ability of field technicians or laboratory analysts to perform routine analyses. The results from this testing may be used for comparison against established criteria or for relative comparisons among the data from a group of technicians or analysts.
Project-specific reporting limit (PSRL) See reporting limit.
Protocol Methodology specified in regulatory, authoritative, or contractual situations.
QC batch The QC batch consists of a set of up to 20 field samples that behave similarly (i.e., same matrix) and are processed using the same procedures, reagents, and standards within the same time period.
QC check sample

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A reference matrix containing known concentrations of parameters of interest. If prepared in the laboratory, it is made using stock standard solutions independent of those used for calibration. If the results of these parameters do not meet acceptance criteria, corrective actions are taken.
Qualification (personnel) The characteristics of abilities gained through education, training, or experience, as measured against established requirements, such as standards or tests, that qualify an individual to perform a required function.
Quality The sum of features and properties/characteristics of a process, item, or service that bears on its ability to meet the stated needs of the user. STL has defined quality as meeting the needs of our clients, both internal and external.
Quality assurance (QA) 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 and quality needed and expected by the customer.
Quality Assurance Directive QA directives are memos issued by the QA Director (or the QA Managers for their facility) to clarify policies, Procedures, and the LQM; or to give direction for an immediate action to ensure or maintain quality.
Quality Assurance Project or Program Plan (QAPP) A formal document describing in comprehensive detail the necessary QA, QC, and other technical activities that must be implemented to ensure the results of the work performed will satisfy the stated performance criteria.
Quality control (QC) The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that it meets the stated requirements established by the client or by STL.
Quality improvement The process of improving the quality of operations. This process encourages worker recommendations for improvement of work processes and requires timely management evaluation and feedback or implementation.
Quality management That aspect of the overall management system of the organization that determines and implements the quality policy. Quality management includes strategic planning, allocation of resources, and other systematic activities (e.g., planning, implementation, and assessment) pertaining to the quality management system.
Quality management system (QMS) A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, and implementation plan of an organization for ensuring quality in its work processes, products, 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.
Random error Variations of repeated measurements that are random in nature and individually not predictable.
Range The difference between the largest and smallest numbers in a set of numbers.

Glossary

Raw data

All documentation associated with the original recording of analytical results pertinent to a specific sample or set of samples. This may include laboratory worksheets, calculation forms, instrument-generated output, analyst notes, etc., from sample receipt through final reporting.

Reagent water

Water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. The reagent water's purity and acceptability is verified by analysis with each set of samples.

Recovery

See percent recovery.

Reference method

A method of known and demonstrated accuracy.

Regression coefficients

The quantities describing the slope and intercept of a regression line.

Relative error

An error expressed as a percentage of the true value or accepted reference value.

Relative percent different (RPD)

Statistic for evaluating the precision of a replicate set. For replicate results:

$$RPD = \left[\frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2} \right)} \right] x\ 100$$

where: X_1 = first observed concentration

X_2 = second observed concentration

Relative response factor (RRF)

$$RRF = \frac{A_x}{A_{IS}} x \frac{C_{IS}}{C_x}$$

A measure of the relative mass spectral response of a compound compared to its internal standard. RRFs are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. Because a RRF is the comparison of two responses, it is a unitless number. RRFs are determined by the following equation:

where: A = area of the characteristic ion measured

C = concentration

IS = internal standard

x = analyte of interest

Relative standard deviation (RSD)

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See coefficient of variation.
Reporting limit (RL) One of two types of reporting limit conventions within STL. The Reporting Limit (RL) is a uniform, STL -wide reporting limit based on an evaluation of the PQLs at STL laboratories and the expected method performance in routine water and soil matrices. Project Specific Reporting Limits (PSRLs) are reporting limits that are defined by project requirements.
Representative sample A sample taken to represent a lot or population as accurately and precisely as possible.
Representativeness Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result (concentration) is representative of the constituent concentration in the sample matrix. At each STL laboratory, every effort must be made to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before subsampling.
Reproducibility The precision, usually expressed as a standard deviation, measuring the variability among results of measurements of the same sample at different laboratories.
Response factor (RF) A factor derived from the calibration of a compound that is used in the quantitation calculation of sample analytes. A response factor may be derived from an external standard calibration (then called a Calibration Factor) or from an internal standard calibration (then called a Relative Response Factor).
Secondary standard A material having a property that is calibrated against a primary standard.
Self assessment Assessments of work conducted by individuals, groups, or organizations directly responsible for overseeing or performing the work.
Shall Denotes a requirement that is mandatory and has to be met.
Should Denotes a guideline or recommendation.
Standard addition The procedure of adding known increments of the analyte of interest to a sample to cause increases in detection response to subsequently establish, by extrapolation of the plotted responses, the level of the analyte of interest present in the original sample.
Standard deviation A measure of the dispersion about the mean of the elements in a population. The square root of the variance of a

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set of values:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$

where: s = standard deviation

Σ = sum of

X = observed values

n = number of observations

standardization The establishment of the value of a potential standard with respect to an established or known standard.
Standard method A method of known and demonstrated precision issued by an organization generally recognized as competent to do so.
Standard operating procedure (SOP) A written document that details an operation, analysis, or action, with prescribed techniques and steps, that is officially approved as the method for performing certain routine or repetitive tasks.
Standard reference material (SRM) A material produced in quantity, of which certain properties have been certified by the National Institute of Standards and Technology (NIST), formerly NBS, or other agencies to the extent possible to satisfy its intended use.
Standard verification Standard is checked by STL or the vendor versus a known specification. See Section 8.5.4.3.
Statistic A constant or coefficient that describes some characteristic of a sample. Statistics are used to estimate parameters of populations.
Stock solution A concentrated solution of analyte(s) or reagent(s) prepared and verified by prescribed procedure(s), and used for preparing working standards or standard solutions.
Subsample A portion taken from a sample. A laboratory sample may be a subsample of a gross sample; similarly, a test portion may be a subsample of a laboratory sample.
Supplier See vendor.
Surrogate (surrogate standard) Compounds, when required by a method, that are used added to every blank, sample, LCS, matrix spike, matrix

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spike duplicate, and standard. They are used to evaluate analytical efficiency by measuring recovery. Surrogates include brominated, fluorinated, or isotopically-labeled compounds that are not expected to be detected in environmental media.
Systematic error The condition of a consistent deviation of the results of a measurement process from the reference or known level.
Systems audit or evaluation A systematic on-site qualitative review of facilities, procedures, equipment, training, record keeping, data verification, and reporting aspects of a quality assurance system to arrive at a measure of the capability of the system. Within STL, system audits or evaluations are performed on a periodic basis under the direction of the STL Corporate Director of Quality Assurance.
Technique Physical or chemical principle for characterizing materials of chemical systems.
Traceability of data The entire documented chain of acquired data from the original acquisition effort through to the final tabulation, synthesis, reduction, and storage activities. The documentation will allow complete reconstruction of the data.
Traceability of samples During all environmental monitoring field efforts, acquired samples will be assigned specific and unique identification numbers. These sample numbers shall be accompanied by documentation (chain-of-custody form) which clearly identifies all parameters associated with sample acquisition. All additional sample numbering systems applied to the sample must be clearly cross-referenced to the field sample number to provide for traceability of samples from acquisition to reporting of sample results.
Traceability of standards The ability of an analytical standard material used for calibration purposes to be traced to its source. The standards used by STL must be traceable via written documentation to sources which produce or sell verified or certified standards, i.e., National Institute for Standards and Technology, or vendors preparing standards from those sources which they have certified.
Validation - computer software The process of establishing documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting predetermined specifications and quality attributes. This process demonstrates and documents that the software performs correctly and meets all specified requirements.
Validation - data The process of a second party performing a systematic review of the raw and final data produced by a laboratory using predetermined criteria to ascertain the validity of the data with respect to the criteria (e.g., HAZWRAP data validation).
Vendor Any individual or organization furnishing items or services or performing work according to a procurement document. This is an all-inclusive term used in place of any of the following: supplier, seller, contractor, subcontractor, or consultant.
Verification - computer software

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The process of checking the accuracy of manually entered or automatically (electronically) calculated information.
Verification - data The process of reviewing data to ensure that data reduction has been correctly performed and that analytical results to be reported correspond to the data acquired and processed.

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QUALITY MANAGEMENT PLAN

Revision: 7

July 2005

Approved by:

President and CEO: _____ Date: 7/28/2005
Rachel Brydon Janetta

Senior Vice President, Chief Operating Officer: _____ Date: 7/28/2005
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SEVERN TRENT STL

Vision

STL will be the recognized industry leader for environmental analysis.

Mission

Through the innovation and dedication of our people, together with the quality of our systems, we will deliver levels of performance that delight our clients, retain the confidence of our stakeholders and enable the profitable growth of our business.

Severn Trent Laboratories

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1. Introduction, Purpose, and Scope

1.1. Severn Trent Laboratories (STL) Overview

Severn Trent Laboratories (STL) is the largest environmental laboratory company in the world with revenues in excess of $300 million and 31 laboratory locations in 19 states nationwide. These facilities have the combined experience of over 500 years in the environmental testing business.

Through continued investment in facilities, equipment, methods and people, STL has developed an unprecedented team of resources, experience and capabilities. With over 2000 chemists, microbiologists and environmental scientists, STL is well positioned to support a variety of clients including commercial, governmental and chemical industries.

STL offers a broad range of environmental testing services. STL's testing capabilities include chemical, physical, and biological analyses of a variety of matrices, including aqueous, solid, drinking water, waste, tissue, air, mold and fungus (Mycology) and saline/estuarine samples. Specialty capabilities include air toxics testing, radiological, mixed waste testing, geotechnical testing, tissue preparation and analysis, aquatic toxicology, dioxin/furan testing, indoor air quality and microscopy services, aquatic toxicology, asbestos analysis, High Resolution Mass Spectrometry (HRMS), Inductively Coupled Plasma/MS (ICP/MS), Liquid Chromatography/MS (LC/MS), PCR microbiology and on-site technologies including mobile laboratories. STL facility locations and contact information are outlined in Table 1.

1.2. Quality Assurance Policy

It is STL's policy to:

- Provide high quality, consistent, and objective environmental testing services that meet all federal, state, and municipal regulatory requirements.
- Generate data that are scientifically sound, legally defensible, meet project objectives, and are appropriate for their intended use.
- Ensure employee adherence to quality documentation and implementation of Corporate Policies and Procedures.
- Provide STL clients with the highest level of professionalism and the best service practices in the industry.
- Build continuous improvement mechanisms into all laboratory, administrative, and managerial activities.
- Maintain a working environment that fosters open communication with both clients and staff and ensures data integrity.

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Table 1 STL Facility Locations

Aerotech (AEL)
4645 East Cotton Center
Blvd., Suite 189
Phoenix, AZ 85040
Phone: 602-437-3340
Fax: 623-445-6192

STL Austin
14050 Summit Drive
Suite A100
Austin, TX 78728
Phone: 512-244-0855
Fax: 512-244-0160

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STL Burlington
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Colchester, VT 05446
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Fax: 802-655-1248

STL Connecticut
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Phone: 203-929-8140
Fax: 203-929-8142

STL Chicago
2417 Bond Street
University Park, IL 60466
Phone: 708-534-5200
Fax: 708-534-5211

STL Corpus Christi
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Phone: 361-289-2673
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STL Denver
4955 Yarrow Street
Arvada, CO 80002
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Fax: 303-431-7171

STL Edison
777 New Durham Road
Edison, NJ 08817
Phone: 732-549-3900
Fax: 732-549-3679

STL Houston
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Houston, TX 77040
Phone: 713-690-4444
Fax: 713-690-5646

STL Knoxville
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Knoxville, TN 37921
Phone: 865-291-3000
Fax: 865-584-4315

STL Los Angeles
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Santa Ana, CA 92705
Phone: 714-258-8610
Fax: 714-258-0921

STL Miami
10200 USA Today Way
Miramar, FL 33025
Phone: 954-431-4550
Fax: 954-431-1959

STL Mobile
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Fax: 251-666-6696

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STL On-Site Technology
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Fax: 413-572-3707

STL Pensacola
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Phone: 850-474-1001
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STL P&K
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STL Pittsburgh
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STL Sacramento
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STL San Francisco
1220 Quarry Lane
Pleasanton, CA 94566-4756
Phone: 925-484-1919
Fax: 925-484-1096

STL Seattle
5755 8th Street East
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13715 Rider Trail North
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Phone: 314-298-8566
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STL Savannah
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1.3. Management Commitment to Quality Assurance and Data Integrity

STL management is committed to providing data of known and documented quality and the best service in the environmental testing industry. To ensure that the data produced and reported by STL meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, STL maintains quality and data integrity systems that are clear, effective, well communicated, and supported at all levels in the company.

1.4. Purpose

The purpose of the Quality Management Plan (QMP) is to describe the STL quality system and to outline how that system enables all employees of STL to meet the Quality Assurance (QA) policy. The QMP also describes specific QA activities and requirements and prescribes their frequencies. Roles and responsibilities of management and laboratory staff in support of the quality system are also defined in the QMP.

1.5. Scope

The requirements set forth in this document are applicable to all STL facilities. Where the document uses the terms “must” and “shall”, this denotes required activities. Practices described in this QMP denote how those activities are performed in general; and each laboratory may have a more detailed description of that activity.

Each STL facility has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where this QMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The facility’s Laboratory Quality Manual (LQM) shall take precedence over the QMP in those cases. Secondarily, each STL facility has the responsibility and authority to operate in compliance with documented client requirements, where they do not conflict with regulatory requirements. STL shall not enter any client agreements that conflict with regulatory requirements in the jurisdiction in which the work is performed. Where documented client agreements conflict with this QMP, but meet the regulatory requirements of the jurisdiction in which the work is performed, the client agreements shall supersede requirements in this QMP.

STL operates under the regulations and guidelines of the following federal programs:

- Air Force Center for Environmental Excellence (AFCEE)
- US Army Corp of Engineers, Hazardous, Toxic and Radioactive Waste (USACE HTRW)
- Clean Air Act (CAA)
- Clean Water Act (CWA)

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- Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
- Department of Energy (DOE)
- Marine Protection, Research, and Sanctuaries Act (MPRSA)
- Navy Facilities Engineering Service Center (NFESC)
- National Pollutant, Discharge, and Elimination System (NPDES)
- Nuclear Regulatory Commission (NRC)
- Occupational Safety and Health Administration (OSHA)
- Resource Conservation and Recovery Act (RCRA)
- Safe Drinking Water Act (SDWA)
- Toxic Substances Control Act (TSCA)

STL also provides services under various state and local municipal guidelines. A listing of each laboratory's service offerings and certifications is presented on STL's website under the MySTL webpage or available from the laboratory.

This QMP was written to comply with the National Environmental Laboratory Accreditation Conference (NELAC) standards. Refer to Table 2 for a cross-section comparison of this QMP to the NELAC standards.

Table 2 Correlation of QMP Sections with NELAC Quality Manual Requirements

NELAC Chapter 5.4.2.3 Quality Manual	Quality Management Plan Section
a. Quality policy statement, including objectives and commitments, by top management	1.2 Quality Assurance Policy 4.2.1 Objectives of the Quality System
b. Organization and management structure	4.1 Organization and Management
c. Relationship between management, technical operations, support services and the quality systems	4.1.2 Roles and Responsibilities 4.2 Quality System
d. Records retention procedures; document control procedures	4.3 Document Control 4.12.2 Record Retention
e. Job descriptions of key staff and references to job descriptions of other staff	4.1.2 Roles and Responsibilities
f. Identification of laboratory approved signatories	4.1 Organization and Management
g. Procedures for achieving traceability of measurements	5.5 Measurement Traceability
h. List of all test methods under which the laboratory performs its accredited testing	5.3.1 Method Selection
i. Mechanisms for assuring the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work	4.4.2 Project-Specific Quality Planning
j. Reference to the calibration and/or verification test procedures used	5.4.3 Equipment Verification and Calibration
k. Procedures for handling submitted samples	4.7.1 Sample Acceptance Policy 5.7 Sample Handling, Transport and Storage
l. Reference to the major equipment and reference measurement standards used as well as the facilities and services used in conducting tests	4.1.1 Laboratory Facilities 5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
m. Reference to procedures for calibration, verification and maintenance of equipment	5.4.2 Equipment Maintenance 5.4.3 Equipment Verification and Calibration
n. Reference to verification practices including inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal QC schemes	5.8.1 Proficiency Testing 5.8.2 Control Samples

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Table 2 Correlation of QMP Sections with NELAC Quality Manual Requirements

NELAC Chapter 5.4.2.3 Quality Manual	Quality Management Plan Section
o. Procedures for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur	4.9 Control of Non-Conformances 4.10 Corrective Action 4.11 Preventive Action 5.8.5 Permitting Departures from Documented Procedures
p. Laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications	4.4.2 Project-Specific Quality Planning 5.8.5 Permitting Departures from Documented Procedures
q. Procedures for dealing with complaints	4.8 Complaints
r. Procedures for protecting confidentiality (including national security concerns) and proprietary rights	4.7.2 Client Confidentiality and Proprietary Rights
s. Procedures for audits and data review	4.13 Internal Audits 4.14 External Audits 5.3.6 Data Reduction and Review
t. Process/procedures for establishing that personnel are adequately experienced in duties they are expected to carry out and are receiving any needed training	5.1.2 Training
u. Reference to procedures for reporting analytical results	5.3.6 Data Review 5.9 Project Reports
v. Table of contents, listing reference, glossaries and appendices	TOC Table of Contents Appendix I: List of Cited SOPs and Work Instructions

2. References

The following references were used in preparation of this document and as the basis of the STL Quality System:

EPA Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA/G-6, US EPA, Office of Environmental Information, March 2001.

EPA Requirements for Quality Management Plans, EPA QA/R-2, US EPA, Office of Environmental Information, March 2001.

EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, US EPA, Office of Environmental Information, March 2001.

EPA Quality Manual for Environmental Programs, 5360 A1, US EPA Office of Environmental Information, Quality Staff, May 2000.

General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025, December 1999.

Good Automated Laboratory Practices, EPA 2185, US EPA, Office of Environmental Information, Resource Management, August 1995.

Quality Assurance Project Plan, HQ Air Force Center for Environmental Excellence, Version 3.1, August 2001.

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National Environmental Laboratory Accreditation Conference Standards, EPA/600/R-04/003, US EPA Office of Research and Development, July 2003.

Navy Installation Restoration Laboratory Quality Assurance Guide, Interim Guidance Document, Naval Facilities Engineering Service Center, February 1996.

Navy Installation Restoration Chemical Data Quality Manual, Navy IR CDQM, September 1999.

Quality Systems Manual for Environmental Laboratories, Department of Defense, Final Version 3, March 2005.

Shell for Analytical Chemistry Requirements, US Army Corps of Engineers, December 1998.

3. *Terms and Definitions*

Accuracy: the degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Audit: a systematic evaluation to determine the conformance to specifications of an operational function or activity.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of 1 to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Chain of Custody (COC): A system of documentation demonstrating the physical possession and traceability of samples.

Clean Air Act: legislation in 42 U.S.C. 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): legislation (42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and reauthorization Act of 1986 (SARA), 42 U.S.C. 9601et seq.

Compromised Sample: a sample received in a condition that jeopardizes the integrity of the results. See Section 4.7.1 for a description of these conditions.

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.

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Confirmation: verification of the presence of a component using an additional analytical technique. These may include second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.

Equipment Blank: a portion of the final rinse water used after decontamination of field equipment; also referred to as Rinsate Blank and Equipment Rinsate.

Document Control: the act of ensuring that documents (electronic or hardcopy 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 is performed.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): legislation under 7 U.S.C. 135 et seq., as amended.

Federal Water Pollution Control Act (Clean Water Act, CWA): legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat. 816.

Field Blank: a blank matrix brought to the field and exposed to field environmental conditions.

Field of Proficiency Testing: NELAC's approach to offering proficiency testing by matrix, technology, and analyte/analyte group.

Good Laboratory Practices (GLP): formal regulations for performing basic laboratory operations outlined in 40 CFR Part 160 and 40 CFR Part 729 and required for activities performed under FIFRA and TSCA.

Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.

Instrument Blank: a blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis.

Internal Chain of Custody: an unbroken trail of accountability that ensures the physical security of samples, data and records. Internal Chain of Custody refers to additional documentation procedures implemented within the laboratory that includes special sample storage requirements, and documentation of all signatures and/or initials, dates, and times of personnel handling specific samples or sample aliquots.

Internal Standard: A standard added to samples in known amount and carried through the procedure as a reference for calibration and controlling instrumental and analytical precision and bias.

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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 (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Laboratory Quality Manual (LQM): a document stating the quality policy, quality system and quality practices of the laboratory. The LQM may include by reference other documentation relating to the laboratory's quality system.

Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory-dependent.

Matrix: the substrate of a test sample. Common matrix descriptions are defined in Table 3.

Matrix Duplicate (MD): duplicate aliquot of a sample processed and analyzed independently; under the same laboratory conditions; also referred to as Sample Duplicate; Laboratory Duplicate.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Matrix Spike Duplicate (MSD): a replicate matrix spike.

Table 3 Matrix Descriptions

Matrix	Description
Air	Air samples as analyzed directly or as adsorbed into a solution or absorption matrix and desorbed.
Aqueous	Aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine source. Includes surface water, groundwater and effluents.
Chemical Waste	A product or by-product of an industrial process that results in a matrix not previously defined.
Drinking Water	Aqueous sample that has been designated a potable water source.
Saline	Aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.
Liquid	Liquid with <15% settleable solids.
Solid	Soil, sediment, sludge or other matrices with ≥15% settleable solids.
Waste	A product or by-product of an industrial process that results in a matrix not previously defined.
Tissue	Sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Method Blank (MB): a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

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Method Detection Limit (MDL): one way to establish a Limit of Detection, defined as the minimum concentration of a substance (an analyte) 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.

Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Precision: an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Preservation: refrigeration and/or reagents added at the time of sample collection to maintain the chemical, physical and/or biological integrity of the sample.

Proficiency Testing: determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.

Proficiency Test (PT) Sample: a sample, the composition of which is unknown to the analyst, that is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. Also referred to as Performance Evaluation (PE) Sample.

Proprietary: belonging to a private person or company.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

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.

Quality Control (QC): the overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control Sample: a sample used to assess the performance of all or a portion of the measurement system. QC samples may be Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking.

Quality Management Plan (QMP): a formal document describing the management policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an agency, organization or laboratory to ensure the quality of its product and the utility of the product to its users.

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/QC.

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Quantitation Limit (QL): the minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the MDL, and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Raw Data: any original information from a measurement activity or study recorded in laboratory notebooks, worksheets, records, memoranda, notes, or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic/optical media, including dictated observations, and recorded data from automated instruments. Reports specifying inclusion of "raw data" do not need all of the above included, but sufficient information to create the reported data.

Record Retention: the systematic collection, indexing and storing of documented information under secure conditions.

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Resource Conservation and Recovery Act (RCRA): legislation under 42 USC 321 et seq. (1976).

Safe Drinking Water Act (SDWA): legislation under 42 USC 300f et seq. (1974), (Public Law 93-523).

Sampling and Analysis Plan (SAP): a formal document describing the detailed sampling and analysis procedures for a specific project.

Selectivity: the capability of a measurement system to respond to a target substance or constituent.

Sensitivity: the difference in the amount or concentration of a substance that corresponds to the smallest difference in a response in a measurement system using a certain probability level.

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

Storage Blank: a blank matrix stored with field samples of a similar matrix.

Systems Audit: a thorough, systematic, on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

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Test Method: an adoption of a scientific technique for performing a specific measurement, as documented in a laboratory SOP or as published by a recognized authority.

Toxic Substances Control Act (TSCA): legislation under 15 USC 2601 et seq., (1976).

Traceability: the property of a result of a measurement that can be related to appropriate international or national standards through an unbroken chain of comparisons.

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.

Verification: confirmation by examination and provision of evidence against specified requirements.

4. Management Requirements

4.1. Organization and Management

4.1.1. Organization

STL's organizational structure is presented in Figure 1. Corporate employees are located at various STL facilities as outlined in the organizational structure. A QA Manager shall be designated at each STL facility.

4.1.2. Roles and Responsibilities

President and CEO
The President of STL, Inc. has overall management responsibility and authority for Severn Trent's laboratory division, including responsibility for budgeting, resource allocation, long term planning, sales, marketing, and final approval on all management and administrative policies and management plans. The President authorizes the QMP and as such, sets the standards for the quality system.

Senior Vice President and Chief Operating Officer (COO)
The COO is responsible for daily management of all STL facilities. The COO's responsibilities include allocation of personnel and resources, long term planning, and development of technical policies and management plans. The COO authorizes the QMP and is responsible for ensuring that business and technical operations are conducted in accordance with its requirements.

Vice President Client and Operations Services (VP COS)
The VP COS is responsible for all essential elements of offerings to clients, including risk management, legal compliance and contract administration, quality assurance, information technology, and environmental health and safety. The VP COS authorizes the QMP and responsibilities include authorization of Manuals, Policies and Procedures, providing support and direction to the Managers of these

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areas, and supporting the COO in decisions regarding long term planning, resource allocation, and capital expenditures.

Corporate Quality Director

The Quality Director is responsible for establishing, implementing and communicating STL's quality system. The Quality Director monitors compliance with the QMP, provides regulatory and technical updates to the STL facilities, assists in development of management plans and technical policies to be approved by the COO, and coordinates training within STL. The Quality Director is available to any employee in STL to resolve data quality or ethical issues. The Quality Director is independent of operational functions.

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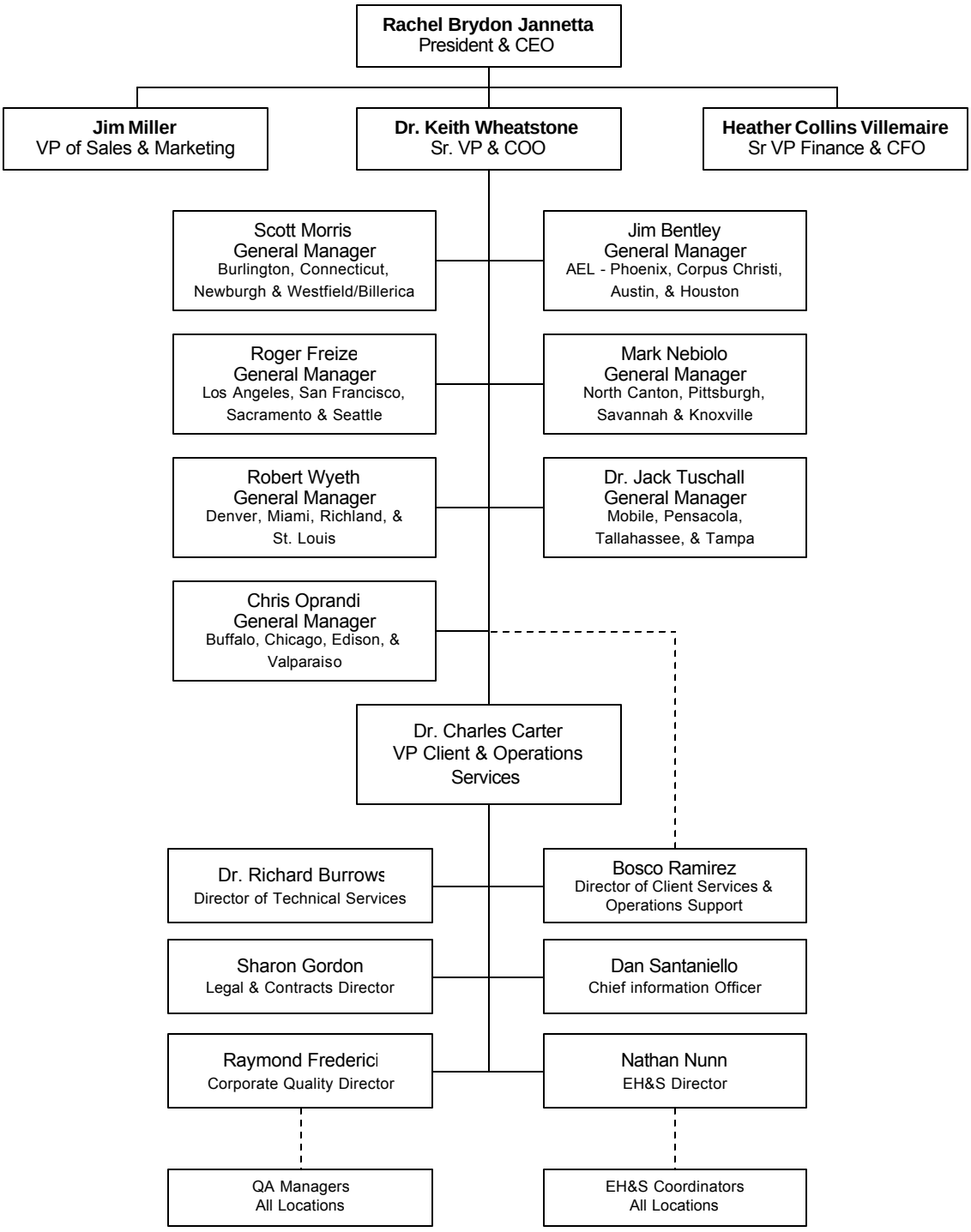
Figure 1. STL Organizational Chart



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Director of Technical Services
The Director of Technical Services is responsible for establishing, implementing and communicating STL's Technical Policies, Standard Operating Procedures, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices throughout STL, advising STL staff on technology advances, innovations, and applications, and organizing and running STL's technical committee.

Director of Client Services and Operations Support
The Director of Client Services and Operations Support is responsible for establishing, implementing and communicating STL's Client Services policies, guidelines and applicable Standard Operating Procedures. The Client Services Directors monitors overall client services indicators and compliance with the appropriate company policies and procedures. The Client Services Director is available to any laboratory to assist in addressing client services issues or to perform reviews and assessments of local client services systems. Other duties include identifying and establishing best client services practices, assisting in Project Management and Client Services training and coordinating STL's Client Service Managers' group.

Chief Information Officer (CIO)
The CIO is responsible for establishing, implementing and communicating STL's Information Technology (IT) Policies, Standard Operating Procedures, and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as STL's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with Good Automated Laboratory Practices (GALP), and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various STL facilities.

Environmental Health and Safety (EH&S) Director
The EH&S Director is responsible for establishing, implementing and communicating STL's Environmental Health and Safety Policies, Standard Operating Procedures, and Manuals. Other responsibilities include conducting EH&S assessments as required, acting as a resource for all STL facilities to ensure EH&S compliance, coordinating safety committees, providing guidance to the EH&S Coordinator at various STL facilities, and advising STL facilities on new EH&S regulations.

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General Manager (GM)
The GM is directly responsible for the daily operations of one or more operating facilities within STL. The GM's responsibilities include allocation of personnel and resources, long term planning, setting goals, and achieving the financial, business, and quality objectives of STL. The GM ensures timely compliance with corporate management directives, policies, and management systems reviews.

Laboratory Director
The Laboratory Director oversees the daily operations of the laboratory. The Laboratory Director's responsibilities include supervision of staff, setting goals and objectives for both the business and the employees, and achieving the financial, business, technical and quality objectives of the facility. The Laboratory Director ensures timely compliance with audits and corrective actions, and is responsible for maintaining a working environment which encourages open, constructive problem solving and continuous improvement.

QA Manager
The QA Manager is responsible for ensuring that the laboratory's quality system and LQM meet the requirements set forth in the QMP, providing quality systems training to all new personnel, maintaining an LQM, and performing or overseeing systems, data, special, and external audits. The QA Manager performs, or supervises, the maintenance of QA records, the maintenance of certifications and accreditations, the submission of monthly QA Reports, and assists in reviewing new work as needed. The QA Manager shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QA Manager is available to any employee at the facility to resolve data quality or ethical issues. The QA Manager shall be independent of laboratory operations. The facility QA Manager has an indirect reporting relationship to the Quality Director. Each LQM has further descriptions of roles and responsibilities at the facility level.

Technical Director
The Technical Director(s) of a laboratory has overall responsibility for a defined portion of the technical operations of the laboratory, and may or may not be the Laboratory Director. The Technical Director solves day to day technical issues, provides technical training and guidance to staff, project managers, and clients, investigates technical issues identified by QA, and directs evaluation of new methods.

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4.2. Quality System

4.2.1. Objectives of STL Quality System

The goal of the STL quality system is to ensure that business and technical operations are conducted with the highest standards of professionalism and ethics in the industry.

To achieve this goal, it is necessary to provide STL clients with not only scientifically sound, well documented, and regulatory compliant data, but also to ensure that STL provides the highest quality service available in the industry with uncompromising data integrity. A well-structured and well-communicated quality system is essential in meeting this goal. STL's quality system is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

The QMP is the basis for STL's quality and data integrity system. It contains requirements and general guidelines under which all STL facilities shall conduct their operations. A table listing the minimum quality system policies and procedures is appended to this QMP. The table includes a citation to the applicable QMP section where a procedure or policy is discussed. It also includes a column indicating the document "Reference".

4.2.2. Laboratory Quality Manual (LQM)

Each STL facility shall have an LQM that further describes the specific QA program at the laboratory.

Each STL facility's LQM shall address:

1. Table of Contents, lists of references and glossaries, and appendices.
2. Quality policy statement, including objectives and commitments, by facility management.
3. Organization and management structure of the laboratory, its place in the STL organization and relevant organizational charts.
4. Relationship between management, technical operations, support services and the quality system.
5. Required Training Elements (e.g., Ethics Training and Technical Training).
6. Record retention procedure.
7. Document control procedure.
8. Job descriptions of essential staff and reference to job descriptions of other staff.
9. Identification of the laboratory's approved signatories.
10. Procedure for achieving traceability of measurements.
11. List of test methods under which the laboratory performs its testing.
12. Procedure for reviewing new work.
13. Reference to the calibration and/or verification test procedures used.
14. Sample handling procedure.
15. Reference to the major equipment, reference standards, facilities and services used by the laboratory in conducting tests.

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16. Reference to procedures for calibration, verification and maintenance of equipment.
17. Reference to verification practices including inter-laboratory comparisons, proficiency testing programs, use of reference materials and internal QC practices.
18. Procedures for feedback and corrective action when testing discrepancies are detected, or departures from policies and procedures occur.
19. Procedure for exceptionally permitting departures from documented policies and procedures or from standard specifications.
20. Procedure for handling client complaints.
21. Procedure for protecting client confidentiality and proprietary rights.
22. Procedure for audits and data review.
23. Procedure for establishing that personnel are adequately experienced and trained.
24. Reference to procedures for reporting analytical results.

4.3. Document Control

4.3.1. Document Type

The following documents, at a minimum, must be controlled at each STL Facility:

- Laboratory Quality Manual
- Standard Operating Procedures (SOP)
- Corporate Quality Management Plan
- Corporate Policies and Procedures

4.3.2. Document Control Procedure

Security and control of documents are necessary to ensure that confidential information is not distributed and that all current copies of a given document are from the latest applicable revision. Unambiguous identification of a controlled document is maintained by identification of the following items in the document header: Document Name, Document Number, Revision Number, Revision Date, Effective Date, Number of Pages. Controlled documents are authorized by Management and/or the QA Department. Controlled documents are marked as such and records of their distribution are kept by the QA Department. Document control maybe achieved by either electronic or hardcopy distribution.

Controlled documents shall be available at all locations where the operational activity described in the document is performed.

4.3.3. Document Revision

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 a revision of the document. When an approved revision of a controlled document is ready for distribution, obsolete copies of the document shall be replaced with the current version of the document. The previous revision of the controlled document must be archived by the QA Department.

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4.3.4. Official Documents

The STL Corporate Operations staff posts Corporate Manuals, Standard Operating Procedures, Policies, Work Instructions, White Papers and Training Materials on STL's intranet site. These are collectively termed "Official Documents" and encompass the Policies and Procedures that all STL facilities are required to employ. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving official documents is found in Corporate SOP S-Q-001, *Official Document Control and Archive*.

4.4. Request, Tender, and Contract Review

4.4.1. Contract Review

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 STL's intent to provide both standard and customized environmental testing services to our clients. To ensure project success, technical staff shall perform a thorough review of technical and QC requirements contained in contracts. Contracts are reviewed for adequately defined requirements and STL's capability to meet those requirements.

Contract review shall include a review of the client's requirements in terms of compound lists, test methodology requested, sensitivity, accuracy, and precision requirements. The STL representative ensures that the laboratory's test methods are suitable to achieve these requirements and must ensure that the laboratory holds the appropriate certifications and approvals to perform the work. The review also includes the laboratory's capabilities in terms of turnaround time, capacity, and resources to provide the services requested, as well the laboratory's ability to provide the documentation, whether hardcopy or electronic. If the laboratory cannot provide all services but intends to subcontract such services, whether to another STL facility or to an outside firm, this must be documented and discussed with the client prior to contract approval.

All contracts entered into by STL shall be reviewed and approved by the appropriate personnel at the facility or facilities performing the work. Any contract requirement or amendment to a contract communicated to STL verbally must be documented and confirmed with the client in writing. Any discrepancy between the client's requirements and STL's capability to meet those requirements is resolved in writing before acceptance of the contract. Contract amendments, initiated by the client and/or STL, are documented in writing for the benefit of both the client and STL.

All contracts, Quality Assurance Project Plans (QAPPs), Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the permanent project record as defined in Section 4.12.1.

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4.4.2. 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, STL assigns a Project Manager (PM) to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively communicated to the laboratory personnel before and during the project.

Each STL facility shall have established project planning procedures in order to ensure that communication is inclusive and effective. These include project memos, designation and meetings of project teams, and meetings between the laboratory staff and the client. STL has found it very effective to invite the client into this process. STL strongly encourages our clients to visit the laboratories and hold formal or informal sessions with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

4.4.3. Data Quality Objectives

Data Quality Objectives (DQO) are qualitative and quantitative statements used to ensure the generation of the type, quantity, and quality of environmental data that will be appropriate for the intended application. Typically, DQOs are identified before project initiation, during the development of QAPPs and SAPs. The analytical DQOs addressed in this section are precision, accuracy, representativeness, completeness, and comparability.

The components of analytical variability (uncertainty) can be estimated when QC samples of the right types and at the appropriate frequency are incorporated into measurement process at the analytical laboratory. STL incorporates numerous QC samples to obtain data for comparison with the analytical DQOs and to ensure that the measurement system is functioning properly. The QC samples and their applications, described in Section 5.8.2, are selected based on regulatory, method- or client-specific requirements. Analytical laboratory QC samples for inorganic, organic, and radionuclide analyses may include calibration blanks, instrument blanks, method blanks, LCS, calibration standards, MS, MSD, surrogate spikes, and yield monitors.

The DQOs discussed below ensure that data are gathered and presented in accordance with procedures appropriate for its intended use, that the data is of known and documented quality, and are able to withstand scientific and legal scrutiny.

Precision is an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions. Precision is expressed either as Relative Standard Deviation (RSD) for greater than two measurements or as Relative Percent

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Difference (RPD) for two measurements. Precision is determined, in part, by analyzing data from aggregate LCS results, MS, MSD, and MD. For radiochemical determinations, counting statistics can also provide an estimate of uncertainty.

Precision also refers to the measurement of the variability associated with the entire process, from sampling to analysis. Total precision of the process can be determined by analysis of duplicate or replicate field samples and measures variability introduced by both the laboratory and field operations.

Accuracy is the degree of agreement between a measurement and the true or expected value, or between the average of a number of measurements and the true or expected value. It reflects the total error associated with a measurement.

Both random and systematic errors can affect accuracy. For chemical properties, accuracy is expressed either as a percent recovery (R) or as a percent bias (R - 100). Accuracy is determined, in part, by analyzing data from LCS, MS, and MSD. For radiochemical determinations, counting statistics can also provide an estimate of uncertainty.

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, a variation in a physical or chemical property at a sampling point, or an environmental condition. Data representativeness is primarily a function of sampling strategy; therefore, the sampling scheme must be designed to maximize representativeness. Representativeness also relates to ensuring that, through sample homogeneity, the sample analysis result is representative of the constituent concentration in the sample matrix. STL makes every effort to analyze an aliquot that is representative of the original sample, and to ensure the homogeneity of the sample before sub-sampling.

Completeness is defined as the percentage of measurements that are judged valid or useable. Factors negatively affecting completeness include the following: sample leakage or breakage in transit or during handling, loss of sample during laboratory analysis through accident or improper handling, improper documentation such that traceability is compromised, or sample result is rejected due to failure to conform to QC specifications. A completeness objective of greater than 90% of the data specified by the statement of work is the goal established for most projects.

Comparability is a measure of the confidence with which one data set can be compared to another. To ensure comparability, all laboratory analysts are required to use uniform procedures (e.g., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

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4.5. Subcontracting

Subcontracting must be arranged with the documented consent of the client, in a timely response which shall not be unreasonably refused. 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. The originating laboratory shall obtain proof of certification from the subcontract facility, and retain this information in STL records. Where applicable, specific QC guidelines, QAPPs, and/or SAPs are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC). It is not acceptable to subcontract work outside of STL without attempting to negotiate alternative requirements with the client and/or the proposed STL subcontract lab.

Non-STL subcontract laboratories may receive an on-site audit by a representative of STL's QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements. The originating laboratory may also perform a paper audit of the subcontractor, which would entail reviewing the LQM, the last two PT studies, and a copy of any recent regulatory audits with the laboratory's responses.

Intra-company subcontracting within STL must be arranged with the documented consent of the client, in a timely response which shall not be unreasonably refused. The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

Project reports from both STL and external subcontractors are discussed in Section 5.9.4.

4.6. Purchasing Services and Supplies

Evaluation and selection of suppliers and vendors is done, 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, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents meet with the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with Corporate SOP S-T-001, *Testing Solvents and Acids*.

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4.7. Service to the Client

4.7.1. Sample Acceptance Policy

Each STL facility shall maintain a sample acceptance policy that describes compromised sample receipt. Samples shall be considered "compromised" if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservative.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal.
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it must be documented in the project records and the client must be contacted for instructions. If the client decides to proceed with the analysis, the project report shall clearly indicate any of the above conditions and the resolution. The PM reviews of daily sample logins must be completed within one business day or prior to sample processing by the laboratory. Specific review criteria are established by each facility to meet specific project needs (e.g., rush samples, hold times, etc..).

4.7.2. Client Confidentiality and Proprietary Rights

Data and sample materials provided by the client or at the client's request, and the results obtained by STL, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay STL for all services rendered or is otherwise in breach of the terms and conditions set forth in the STL and client contract) subject to any disclosure required by law or legal process. STL's reports, and the data and information provided therein, are for the exclusive use and benefit of the client, and are not released to a third party without written consent from the client.

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4.8. Complaints

STL believes that an effective client complaint handling process has important business and strategic value. Listening to and documenting client's concerns captures "client knowledge" that helps to continually improve the process and outpace the competition. Implementing a client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

Client complaints shall be documented, communicated to management, and addressed promptly and thoroughly. Client complaints are documented by the employee receiving the complaint. The documentation can take the form of a corrective action report (as described in Section 4.10) or in a format specifically designed for that purpose. The Laboratory Director, PM, Customer Service Manager, and QA Manager are informed of all client complaints, and assist in resolving the complaint.

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 is required to conduct a special audit to assist in resolving the issue. A written confirmation, or letter to the client, outlining the issue and response taken is strongly recommended as part of the overall action taken.

The number and nature of client complaints shall be reported to the Quality Director in the QA Monthly report submitted by each facility. The overall number of complaints received per facility is tracked and the appropriateness of the response to client complaints is assessed. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Management Systems Review. Most client feedback comes either verbally or in writing to STL employees. However, STL also uses a number of additional mechanisms to obtain client feedback including the ACIL Seal of Excellence survey, a biannual customer satisfaction survey, and a response card system. Each of these is monitored for trends and opportunities for improvement.

4.9. Control of Non-conformances

Each STL facility shall have a procedure to control and document non-conformances. Non-conformances include any out of control occurrence. Non-conformances may relate to client specific requirements, procedural requirements, or equipment issues. All non-conformances in the laboratory are documented at the time of their occurrence.

All non-conformances that affect a sample and/or sample data become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If

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the reanalysis comes back within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, Manager, PM, Laboratory Director, or QA Manager for direction may be required. All records of reanalysis are kept with the project files.

Where non-conformances specifically affect a client's sample and/or data, the client shall be informed and action must be taken. Action can take the form of reporting and flagging the data, and including a description of the non-conformance in the project narrative or cover letter.

4.10. Corrective Action

4.10.1. General

Each STL facility shall maintain an established, documented corrective action process. Each corrective action is thoroughly investigated, and the investigation, outcome of the investigation, action taken, and follow-up is documented. Corrective action reports are reviewed, approved, and maintained by the QA department.

4.10.2. Initiation

Any employee in STL shall be authorized to initiate a corrective action. The initial source of corrective action can also be external to STL (e.g., corrective action due to client complaint, regulatory audit, or proficiency test). When a problem that requires corrective action is identified, the following items are identified by the initiator on the corrective action report: the nature of the problem, the name of the initiator, and the date. If the problem effects a specific client project, the name of the client and laboratory project number is recorded, and the PM is informed immediately.

4.10.3. Cause Analysis

The corrective action process must be embarked upon as a joint, problem solving, constructive effort. Identification of systematic errors, or errors that are likely to occur repetitively due to a defect or weakness in a system, is particularly valuable in maintaining an environment of continuous improvement in laboratory operations.

When a corrective action report is initiated, the initiator works with the affected employee(s) and/or department(s) to identify the root cause of the problem. An essential part of the corrective action process is to identify whether the problem occurred due to a systematic or isolated error.

If the initiator of the corrective action report is uncertain as to what would constitute appropriate corrective action or is unable to resolve the situation, the problem is identified to the Supervisor, Manager, Laboratory Director or the QA Manager who

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provides assistance in the corrective action process. The root cause of the problem and associated cause analysis is documented.

4.10.4. Corrective Action

Once the root cause of a problem is identified, the initiator and affected employee(s) and/or department(s) examine potential actions that will rectify the present problem to the extent possible, and prevent recurrence of future, similar occurrences. An appropriate corrective action is then recommended. The corrective action must be appropriate for the size and nature of the issue.

If the corrective action concerns a specific project related issue, the PM or Customer Service Manager approves the corrective action before its implementation.

Implementation of the corrective action and the date of implementation are documented on the corrective action report.

If a corrective action is related to a specific project report, it is included in the project file. An essential part of the corrective action process is communication and awareness of the problem, the cause, and the action taken to prevent future occurrences and/or rectify the immediate problem.

4.10.5. Monitoring Corrective Action

The QA department reviews corrective action reports and selects one or more of the more significant corrective actions for inclusion in the annual systems audit. The QA Department also may implement a special audit. The purpose of inclusion of the corrective action process in both routine and special audits is to monitor the implementation of the corrective action and to determine whether the action taken has been effective in overcoming the issue identified.

4.11. Preventative Action

Each STL facility shall maintain an established, documented preventative action process. Preventative action is identifying process weaknesses which have the potential to lead to failure(s). Preventative action includes analysis of the quality system to detect, analyze, and eliminate potential causes of non-conformances. When potential problems are identified, preventative action is initiated to effectively address the problem to eliminate or reduce the risk identified.

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4.12. Records

4.12.1. Record Types

Table 4 STL Record Types[1]

Raw Data	Controlled Documents	QA Records	Project Records	Administrative Records
See Section 3. Terms and Definitions	LQM	Audits/ Responses	COC Documentation	Accounting
	QMP	Certifications	Contracts and Amendments	EH&S Manual, Permits, Disposal Records
	SOPs	Corrective Action	Correspondence	Employee Handbook
		Logbooks[2]	QAPP	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)
		Method & Software Validation, Verification data	SAP	
		Standards Certificates	Telephone Logbooks	Technical and Administrative Policies
		Technical Training Records		

[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).

4.12.2. Record Retention

Table 5 outlines STL's standard record retention time. For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled 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 6 have lengthier retention requirements and are subject to the requirements in Section 4.12.3.

Table 5 STL Record Retention

Record Type[1]		Archival Requirement
Raw Data	All*	5 Years from analytical report issue
Controlled Documents	All*	5 Years from document retirement date
QA	All*	5 Years from archival
Project	All*	5 Years from analytical report issue
Administrative	Personnel/Training	7 years
	Accounting	See Accounting and Control Procedures Manual

[1] Record Types encompass hardcopy and electronic records.
* Exceptions listed in Table 6.

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Table 6 Special Record Retention Requirements

Program	[1]Retention Requirement
Colorado – Drinking Water	10 years
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Minnesota – Drinking Water	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
NY Potable Water NYCRR Part 55-2	10 years
OSHA - 29 CFR Part 1910	30 years
Pennsylvania – Drinking Water	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

[1]Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

4.12.3. Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the STL standard record retention time. These are detailed in Table 6 with their retention requirements. In these cases, the longer retention requirement must be implemented and noted in the archive or addressed in a facility specific records retention procedure. 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.

4.12.4. Archives and Record Transfer

Archives must be indexed such that records are accessible on either a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented. On-site and/or off-site facilities may be used.

STL ensures that all records are maintained as required by the regulatory guidelines and per the QMP upon facility location change or ownership transfer. Upon STL facility location change, all archives are retained by STL in accordance with the QMP. Upon ownership transfer, record retention requirements shall be

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addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established.

4.13. Internal Audits

4.13.1. Audit Types and Frequency

A number of types of audits shall be performed at STL. Audit type and frequency are categorized in Table 7.

Table 7 Audit Types and Frequency

Audit Type	Performed by	Frequency
Systems	QA Department or Designee	Annual
Data	QA Department or designee	**Data Report Review:** As necessary to ensure an effective secondary review process
		Analyst Data Audits: 100% of all analysts annually
		Electronic Data Audits: 100% of all organic instruments
Special	QA Department or Designee	As Needed

4.13.2. Systems Audits

Facility systems audits are technical in nature and are conducted on an ongoing basis by the QA Manager or his/her designee at each facility. Systems audits cover all departments of the facility, both operational and support.

The audit report is issued by internal auditor within 30 calendar days of the audit. The audit report is addressed to the Laboratory Director, and copied to the Corporate Quality Director and General Manager. If the internal audit is performed by someone other than the facility's QA Manager, the report must also be addressed to the QA Manager.

Written audit responses are required within 30 calendar days of the audit report issue. The audit response follows the format of the audit report, and corrective actions and time frames for their implementation are included for each deficiency. The audit response is directed to all individuals copied on the audit report. Where a corrective action requires longer than 30 days to complete, the target date for the corrective action implementation is stated and evidence of the corrective action is submitted to the QA Department in the agreed upon time frame.

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4.13.3. Data Audits

Data audits assess the level of customer service, SOP compliance, regulatory compliance, accuracy and completeness of test results and reports, documentation, and adherence to established QC criteria, laboratory SOPs, technical policy, and project specific QC criteria. Data Audits may be accomplished through electronic instrument data audits, analyst data authenticity audits or through final project report reviews.

Records of the data audits shall be kept, and the frequency of data audits shall be included in the monthly QA report. In performing data audits, it is essential that data be assessed in terms of differentiating between systematic and isolated errors. Upon noting anomalous data or occurrences in the data audits, the QA Department is responsible for seeking clarification from the appropriate personnel, ascertaining whether the error is systematic or an isolated error, and overseeing correction and/or revision of the project report if necessary. Errors found in client project reports are revised and the revision sent to the client. The QA Department is also responsible for assisting in the corrective action process where a data audit leads to identification of the need for process evaluation and change.

Where specific clients and regulatory programs require more frequent data auditing, the individual facility must meet the data auditing frequency for that program.

4.13.3.1. Data Authenticity Audits

Data authenticity audits shall be performed on 100% of all analysts by the QA department or a designee independent from the operations. Performing data authenticity checks will typically include verifying raw data, evaluating calculation tools and independently reproducing the final results and comparing it to the hardcopy on randomly selected batches of data. Analyst data audits must include spot-checking of manual integrations by QA personnel in order to determine that the manual integration is appropriate and documented according to Section 5.3.6. The laboratory will report the percentage of analysts reviewed (for the year) in their monthly QA report and should average about 8% per month.

4.13.3.2. Electronic Data Audits

Electronic data audits shall be performed on 100% of all organic instruments by the QA department or a designate independent from the operations. This may include Mint Miner® scanning of randomly selected batches of electronic data followed by a chromatography system review. The laboratory will report the percentage of instruments reviewed (for the year) in their monthly QA report and should average about 8% of instruments per month. Electronic data audits must include spot-checking of manual integrations by QA personnel in

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order to determine that the manual integration is appropriate and documented according to Section 5.3.6.

4.13.3.3. Final Report Reviews

The frequency of auditing final reports depends on the effectiveness of the laboratory's secondary review process. If the laboratory infrequently finds report errors or there is a low percentage of revised reports due to analytical error, audits may be less frequent.

4.13.4. 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, proficiency testing results, data audits, systems audits, validation comments, or regulatory audits. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

4.14. External Audits

STL facilities are routinely audited by clients and external regulatory authorities. STL is available for these audits and makes every effort to provide the auditors with the personnel, documentation, and assistance required by the auditors. STL recommends that the audits be scheduled with the QA Department so that all necessary personnel are available on the day of the audit.

4.15. Management Reviews

4.15.1. QA Reports to Management

A monthly QA report shall be prepared by the QA Manager or their designee and forwarded to the Laboratory Director and the Quality Director. The reports include statistical results that are used to assess the effectiveness of the quality system. At a minimum, the content of the monthly report is shown in Figure 2.

A Corporate QA Monthly Report containing a compilation of the Facility QA reports statistics, information on progress of the Corporate QA program, and a narrative outlining significant occurrences and/or concerns shall be prepared by the Quality Director and forwarded to the General Manager of Operational and Technical Services and the COO.

4.15.2. Management Systems Review

Each STL facility shall perform a management quality system review annually in accordance with the corporate standard operating procedure. This will synchronize quality planning with fiscal year planning. The management quality system will assess the adequacy of the laboratory's quality system and plan any changes in

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laboratory organization, policies, practices, certifications, accreditations in order to achieve operational efficiencies, meet regulatory requirements and client expectations.

4.15.3. Monthly QA Report and Metrics

Each laboratory's QA Manager will prepare a monthly QA report which is due by the third working day of the month. The report will be sent to the Laboratory Director, General Manager and Quality Director. The report will contain a narrative summary and metrics spreadsheet. At a minimum, the report content will contain the items listed below. During the course of the year, the Laboratory Director, General Manager or Quality Director may request that additional information be added to the report.

Figure 2 Monthly QA Report Format

1	**Audits**
	Internal System Audits External System Audits
2	**Revised Reports**
	Revised Reports Corrective/Preventive action measures
3	**Client Complaints**
	Describe situations and resolutions in progress
4	**Certifications/Approvals**
	Issues/changes Lapses/potential revocations
5	**Proficiency Testing**
	Study participation and scores Combined PT scores Repeat failures
6	**SOP Status**
	Report the percentage of SOPs that have been revised or reviewed within the last 24 months
7	**Miscellaneous QA and Operational Issues**
	Narrative outlining improvements, regulatory compliance issues and general concerns
Appended	**Metrics Spreadsheet**
	Summarize metrics in the template provided by the Corporate Quality Director

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5. Technical Requirements

5.1. Personnel

5.1.1. General

STL management believes that its highly qualified, ethical and professional staff is the single most important aspect in assuring the highest level of data quality and service in the industry.

STL staff consists of over two thousand professionals and support personnel that include the following positions:

- General Manager
- Customer Service Manager
- Quality Assurance (QA) Manager
- Laboratory Director
- Technical Director
- Laboratory Manager
- Department Supervisor
- Information Technology Manager
- Human Resources Manager
- Project Manager
- Department Manager
- Analyst
- Sample Custodian
- Technician
- Quality Assurance Specialist
- Data Review Specialist
- Information Technology Specialist

5.1.2. Training

STL is committed to furthering the professional and technical development of employees at all levels. Minimum training requirements for STL employees are outlined in Table 8.

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Table 8 STL Employee Minimum Training Requirements

Required Training	Time Frame*	Employee Type
Environmental Health & Safety	Month 1	All
Ethics – New Hires	1-2 days of hire	All
Ethics - Comprehensive Data Integrity Quality Assurance	30 days of hire (All training)	All Technical and PMs All
Ethics Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

**From date of initial employment unless otherwise indicated.*

Technical training is accomplished within each laboratory by management to ensure method comprehension. All new personnel shall be required to demonstrate competency in performing a particular method by successfully completing an Initial Demonstration of Capability (DOC) before conducting analysis independently on client samples.

DOCs are performed by analysis of four replicate QC samples. Results of successive LCS analyses can be used to fulfill the DOC requirement. The accuracy and precision, measured as average recovery and standard deviation (using n-1 as the population), of the 4 replicate results are calculated and compared to those in the test method (where available). If the test method does not include accuracy and precision requirements, the results are compared to target criteria set by the laboratory. The laboratory sets the target criteria such that they reflect the DQOs of the specific test method or project. A DOC Certification Statement is recorded and maintained in the employee's training or personnel file. Figure 3 shows an example of a DOC Certification Statement.

The following evidence must be on file at the laboratory for each technical employee:

- DOC.
- The employee has read and understood the latest version of the laboratory's quality documentation.
- The employee has read and understood the latest, approved version of all test methods and/or SOPs for which the employee is responsible.
- Annual evidence of continued DOC that may include successful analysis of a blind sample on the specific test method, or a similar test method, or an annual DOC, or four successive, successful LCSs.
- An Ethics Agreement signed by each staff member (renewed each year).
- A Confidentiality Agreement signed by each staff member (renewed each year).

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Figure 3 Example Demonstration of Capability Certification Statement

Demonstration of Capability
Certification Statement

Date: Matrix:
Laboratory Name: Method:
Laboratory Address:
Analyst Name:

We the undersigned certify that:

1. The analyst identified above, using the cited test method, which is in use at this facility for the analysis of samples under the National Environmental Laboratory Accreditation Program, has met the Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification.
3. Copies of the test method and SOP are available for all personnel on site.
4. The data associated with the DOC are true, complete and representative.
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is available for review by authorized inspectors.

Laboratory Manager/Supervisor _____ Signature _____ Date _____

5.1.3. Ethics Policy

Establishing and maintaining a high ethical standard is an important element of a quality system. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times, STL has established an *Ethics Policy*, P-L-006, and an Ethics Agreement (Figure 4). Each employee shall sign the Ethics Agreement, signifying agreed compliance with its stated purpose. The ethics agreement is required to be re-signed on an annual basis.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize the Company's ability to do work on Government contracts, and for that reason, the Company has a Zero Tolerance approach to such violations.

Ethics is also a major component of STL's quality and data integrity systems. Each employee must be introduced to STL's Ethics Policy within 1-2 days of hire; and receive the Comprehensive Ethics, Data Integrity Training and Quality Training within 30 days of hire. Annually, Ethics Refresher Training will be provided. Employees must be trained as to the legal and environmental repercussions that result from data misrepresentation. A data integrity hotline is maintained by STL and administered by the Quality Director.

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Figure 4 STL Ethics Agreement

I understand that STL is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.

With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:

- I will not intentionally report data values that are not the actual values obtained;
- I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations;
- I will not intentionally misrepresent another individual's work;
- I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers;
- I agree to inform my Supervisor of any accidental reporting of non-authentic data by me in a timely manner; and I agree to inform my Supervisor of any accidental or intentional reporting of non-authentic data by other employees;
- If a supervisor or a member of STL management requests me to engage in or perform an activity that I feel is compromising data validity or quality, I will not comply with the request and will report this action immediately to a member of senior management, up to and including the President of STL; and
- I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the Severn Trent family of companies.

As a STL employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.

The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.

EMPLOYEE SIGNATURE _____ Date _____

Supervisor/Trainer: _____ Date _____

5.2. Facilities

Each STL facility must be secure and access must be controlled and documented. Access is controlled by various measures including locked doors, passwords, electronic access cards, security codes, and staffed reception areas. All visitors sign in and are escorted by STL personnel while at an STL facility.

STL's facilities are designed for efficient, automated high-quality operations. All laboratories are equipped with Heating, Ventilation, and Air Conditioning (HVAC) systems appropriate to the needs of environmental testing laboratories. Environmental conditions in the facilities, such as hood flow, are routinely monitored and documented. Table 9 summarizes the square footage at each STL facility.

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All STL facilities are 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. STL also provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, respirators, etc.

Table 9 STL Laboratory Square Footage

Facility	Square Footage
STL Austin	27,000
STL Billerica	3,5,000
STL Buffalo	32,000
STL Burlington	36,000
STL Chicago	48,500
STL Connecticut	17,000
STL Corpus Christi	14,000
STL Denver	54,000
STL Edison	42,000
STL Houston	28,000
STL Knoxville	29,000
STL Los Angeles	27,000
STL Miami	17,000
STL Mobile	14,000
STL Newburgh	8,000

Facility	Square Footage
STL North Canton	53,000
STL Pensacola	25,000
STL Pittsburgh	30,000
STL Richland	33,000
STL Sacramento	66,000
STL Savannah	55,000
STL San Francisco	21,000
STL Seattle	20,000
STL St. Louis	31,000
STL Tallahassee	22,000
STL Tampa	14,000
STL Valparaiso	14,500
STL Westfield	10,000
AEL, Phoenix	24,000

5.3. Test Methods

5.3.1. Method Selection

Most of the test methods performed at STL originate from test methods published by a regulatory agency such as the U.S. EPA and other state and federal regulatory agencies. These include, but are not limited to, the following published compendiums of test methods:

Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999

Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, August 1980.

Eastern Environmental Radiation Facility Radiochemistry Procedures Manual, EPA, PB84-215581, June 1984.

HASL-300 28th Edition, Environmental Measurements Laboratory (EML), 1997.

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Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fourth Edition, EPA/600/4-90/027F, August 1993.

Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, EPA-821-R-02-012, October 2002.

Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, Fourth Edition, EPA-821-R-02-013, October 2002.

Analytical Method for Determination of Asbestos Fibers in Water, EPA-600/4-83, September 1983.

Determination of Asbestos Structures Over 10-mm in Length in Drinking Water, EPA-600/R-94-134, June 1994.

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.

Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water.

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.

NIOSH Manual of Analytical Methods, 4th ed., August 1994.

Statement of Work for Inorganics Analysis, ILM04.1, USEPA Contract Laboratory Program Multi-media, Multi-concentration.

Statement of Work for Organics Analysis, OLM04.2, USEPA Contract Laboratory Program, Multi-media, Multi-concentration.

Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLMO4.1, USEPA Contract Laboratory Program, September 1998.

Standard Methods for the Examination of Water and Wastewater, 18th/19th /20th 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.

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Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.

National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.

5.3.2. SOPs

Each STL facility shall maintain an SOP Index for both Method and Process SOPs. Method SOPs are maintained to describe a specific test method. Process SOPs are maintained to describe functions and processes not related to a specific test method.

Method SOPs contain the following information:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 5).

Identification of Test Method
Applicable Matrix
Reporting Limit
Scope and Application, including test analytes
Summary of the Test Method
Definitions
Interferences
Safety
Equipment and Supplies
Reagents and Standards
Sample Collection, Preservation, Shipment and Storage
Quality Control

Calibration and Standardization
Procedure
Calculations
Method Performance
Pollution Prevention
Data Assessment and Acceptance Criteria for Quality Control Measures
Corrective Actions for Out-of-Control Data
Contingencies for Handling Out-of-Control or Unacceptable Data
Waste Management
References
Tables, Diagrams, Flowcharts and Validation Data

Process SOPs may contain the following information:

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 5).

Scope
Summary
Definitions
Responsibilities

Safety
Procedure
References
Tables, Diagrams and Flowcharts

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The QA Department is responsible for maintenance of SOPs, archival of SOP historical revisions, maintenance of an SOP index, and records of controlled distribution. SOPs, at a minimum, must undergo periodic review as described the each facility's LQM or SOP. Where an SOP is based on a published method, the laboratory must maintain a copy of the reference method.

Figure 5 Proprietary Information Statement

This documentation has been prepared by Severn Trent Laboratories (STL) solely for STL's own use and the use of STL's customers in evaluating its qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to STL upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

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SOP Appendix

In some cases, a standard laboratory procedure is modified slightly for a specific client or project at the client or regulatory agency's request. In these cases, an Appendix to the SOP may be attached that indicates the modifications to the SOP which are specific to that project. SOP appendices shall not be used to alter test methods required by regulation such that the modifications would result in non-compliance with the regulation.

5.3.3. Method Validation

Laboratory developed methods are validated and documented according to the procedure described in Section 5.3.5.

5.3.4. Method Verification

Method verification is required when a validated standard test method or a method modification is implemented. The level of activity required for method verification is dependent on the type of method being implemented, or on the level of method modification and its affect on a method's robustness. Method modification often takes advantage of a method's robustness, or the ability to make minor changes in a method without affecting the method's outcome. Method verification may require some, but not all, of the activities described in Section 5.3.5.

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5.3.5. Method Validation and Verification Activities

Before analyzing samples by a particular method, method validation and/or method verification must occur. A complete validation of the method is required for laboratory developed 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.

Determination of Method Selectivity
Method selectivity is demonstrated for the analyte(s) in the specific matrix or matrices. In some cases, to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

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. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed. Each STL facility must document their approach to estimation and/or demonstration of sensitivity. Refer to the Corporate SOP S-Q-003, *Method Detection Limit Studies*, additional information.

Relationship of the Limit of Detection (LOD) to the Quantitation Limit (QL)

Each laboratory shall have a procedure to relate the QL to the LOD (or MDL if appropriate). An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum level at which both the presence of an analyte and its concentration can be reliably determined. 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 QL. 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 QL, 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 reported with a qualification that denotes the semi-quantitative nature of the result.

Determination of Interferences
A determination that the method is free from interferences in a blank matrix is performed.

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Determination of Range
Where appropriate, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

Initial Demonstration of Capability
DOCs are performed prior to method performance.

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.

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 Appendix describing the specific differences in the new method is acceptable in place of a separate SOP.

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 LCSs and Method Blanks.

5.3.6. Data Review

All data, regardless of regulatory program or level of reporting, shall be subject to a thorough review which involves a primary, secondary, and completeness review process. All levels of the review must be documented.

Primary Review

The primary review is often referred to as a “bench-level” review. In most cases, the analyst who generates the data (e.g., logs in, prepares and/or runs the samples) is the primary reviewer. In some cases, an analyst may be reducing data for samples run by an auto-sampler set up by a different analyst. In this case, the identity of both the analyst and the primary reviewer is identified in the raw data.

One of the most important aspects of primary review is to make sure that the test instructions are clear, and that all project specific requirements have been understood and followed.

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Once an analysis is complete, the primary reviewer must ensure that:

- Sample preparation information is complete, accurate, and documented.
- Calculations have been performed correctly.
- Quantitation has been performed accurately.
- Qualitative identifications are accurate.
- Manual integrations are appropriate.
- Data flags to indicate manual integrations are recorded.
- Manual integrations are authorized by a date and signature or initials (hardcopy or electronic) of primary analyst.
- Client specific requirements have been followed.
- Method and process SOPs have been followed.
- Method QC criteria have been met.
- QC samples are within established limits.
- Dilution factors are correctly recorded and applied.
- Non-conformances and/or anomalous data have been properly documented and appropriately communicated.
- COC procedures have been followed.
- All unused portions of hardbound logbooks are 'Z'ed out; corrections are made with a single line drawn through the error and are dated and initialed.
- Primary review is documented by date and initials/signature of primary analyst.

Any anomalous results and/or non-conformances noted during the Primary Review are documented on a data review checklist (defined by each facility) communicated to the Supervisor and the PM for resolution. Resolution can require sample reanalysis, or it may require that data be reported with a qualification. Non-conformances are documented per Section 4.9.

Secondary Review

The secondary review shall be a complete technical review of a data set. The secondary review must be documented and the secondary reviewer identified. The following items are reviewed:

- Qualitative Identification
- Quantitative Accuracy
- Calibration
- QC Samples
- Method QC Criteria
- Adherence to method and process SOPs
- Accuracy of Final Client Reporting Forms

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- Manual Integrations – Minimal requirement is to spot-check raw data files for manual integration, as verified by date and initials or signature (hardcopy or electronic) of secondary data reviewer. Some regulatory programs require 100% secondary review of manual integrations.
- Completeness
- Special Requirements/Instructions

If problems are found during the secondary review, which are documented on the data review checklist (defined above), the reviewer must work with the appropriate personnel to resolve them. If changes are made to the data, such as alternate qualitative identifications, identifications of additional target analytes, re-quantitation, or re-integration, the secondary reviewer must contact the laboratory analyst and/or primary reviewer of the data so that the primary analyst and/or reviewer is aware of the appropriate reporting procedures.

Completeness Review

The completeness review shall include the generation of a project narrative and/or cover letter which outlines anomalous data and non-compliances using project narrative notes and non-compliance reports generated during the primary and secondary review. The completeness review addresses the following items:

- Is the project report complete?
- Does the data meet with the client's expectations?
- Were the data quality objectives of the project met?
- Are QC outages and/or non-conformances approved and appropriately explained in the narrative notes?

5.3.7. Data Integrity and Security

This section details those procedures that are relevant to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data.

Security and Traceability

Access to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data must be both controlled and recorded. There are various systems at STL to which this applies, which include the Laboratory Information Management System (LIMS), as well as specific systems such as chromatography data systems.

Control of the system is accomplished through limitation of access to the system by users with the education, training and experience to perform the task knowledgeably and accurately. System users are granted privileges that are commensurate with their experience and responsibilities.

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Computer access is tracked by using unique login names and passwords for all employees that have access to the computer system. "General" or "multi-user" account access to computer systems that collect, analyze and process raw instrumental data, and those that manage and report data shall not be permitted. Entries and changes are documented with the identity of the individual making the entry, and the time and date. Where a computer system is processing raw instrumental data, the instrument identification number as described in Section 5.4.1 is recorded. Many of these systems have the capability of maintaining audit trails to track entries and changes to the data. This function is activated on any computer system that has that capability.

STL requires that all sensitive computer systems, defined as LIMS servers and other servers of critical importance, be locked in a secured room. Access must be limited only to employees who need physical access to those systems. This room must also provide climate control within the parameters provided by the vendor of the secured equipment.

Verification

All commercially obtained software shall be verified prior to use and after version upgrade. Verification involves assessing whether the computer system accurately performs its intended function. Verification generally is accomplished by comparing the output of the program with the output of the raw data manually processed, or processed by the software being replaced. The records of the verification are required to contain the following information: software vendor, name of product, version, comparison of program output and manual output, raw data used to verify the program, date, and name of the individual performing the verification. Records of verification are retained as QC records.

Validation

Software validation involves documentation of specifications and coding as well as verification of results. Software validation is performed on all in-house programs. Records of validation include original specifications, identity of code, printout of code, software name, software version, name of individual writing the code, comparison of program output with specifications, and verification records as specified above. Records of validation are retained as QC records.

Auditing

The QA Departments system audits includes review of the control, security, and tracking of IT systems and software.

Version Control

The laboratory shall maintain copies of outdated versions of software and associated manuals for all software in use at the laboratory for a period of five years from its retirement date. The associated hardware, required to operate the software, must also be retained for the same time period.

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5.4. Equipment

5.4.1. Equipment Operation

STL is committed to routinely updating and automating instrumentation. STL facilities maintain state of the art instrumentation to perform the analyses within the QC specifications of the test methods. Each STL facility shall maintain an equipment list that must include the following information:

- Date Installed or year placed in service
- Manufacturer's Name, Model Number, Serial Number
- Current Location
- Preventative Maintenance Schedule

All equipment is subject to rigorous checks upon its receipt, upgrade, or modification to establish that the equipment meets with the selectivity, accuracy, and precision required by the test method for which it is to be used. All manufacturer's operations and maintenance manuals are kept up to date and accessible for the use of the equipment operator. Documentation of equipment usage is maintained using analytical run and maintenance logbooks or the electronic versions of said documents.

5.4.2. Equipment Maintenance

Each STL facility must employ a system of preventative maintenance in order to ensure system up time, minimize corrective maintenance costs and ensure data validity. All routine maintenance is performed as recommended by the manufacturer and may be performed by an analyst, instrument specialist or outside technician. Maintenance logbooks or electronic records are kept on all major pieces of equipment in which both routine and non-routine maintenance is recorded. Notation of the date and maintenance activity is recorded each time service procedures are performed. The return to analytical control following instrument repair is documented. Maintenance logbooks or electronic records are retained as QA records.

Maintenance contracts are held on specific pieces of equipment where outside service is efficient, cost-effective, and necessary for effective operation of the laboratory.

5.4.3. Equipment Verification and Calibration

All equipment shall be tested upon receipt to establish its ability to meet the QC guidelines contained in the test method for which the instrumentation is to be used. This testing shall be documented. Once an instrument is placed in routine service, ongoing instrument calibration is demonstrated at the appropriate frequency as defined in the test method. Refer to the Corporate Policy P-T-001, *Selection of Calibration Points*, for guidance on using calibration data. Any instrument that is

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deemed to be malfunctioning is clearly marked and taken out of service. When the instrument is brought back into control, acceptable performance is documented.

5.5. Measurement Traceability

5.5.1. General

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. With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), 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.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

Laboratory DI and RO water systems have documented preventative maintenance schedules and the conductivity of the water is recorded on each day of use.

5.5.2. Reference Standards Traceability

The receipt of all reference standards must be documented. ~~References~~ 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 standards should be purchased with an accompanying Certificate of Analysis that documents the standard 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 documentation of standard purity is archived, and references the Standard Identification Number.

All efforts are made to purchase standards that are $\geq$ 97.0% purity or as prescribed by the methods. If this is not possible, the purity is used in performing standards calculations.

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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 different lot is acceptable for use as 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 is used as the second source confirmation.

5.5.3. Reagents

Reagents are, in general, required to be analytical reagent grade unless otherwise specified in method SOPs. Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented.

5.6. Sampling Plans

Sample representativeness and integrity are the foundations upon which meaningful analytical results rely. Where documented and approved SAPs and/or QAPPs are in place, they must be made available to the laboratory before sample receipt, and approved by laboratory management before sample receipt.

5.7. Sample Handling, Transport, and Storage

5.7.1. General

Chain of Custody (COC) can be established either when bottles are sent to the field, or at the time of sampling. STL can provide all of the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory.

Samples are received at the laboratory by a designated sample custodian and a unique Laboratory Project Identification Number is assigned. The following information is recorded for each sample shipment: Client/Project Name, Date and Time of Laboratory Receipt, Laboratory Project Number, and Signature or initials of the personnel receiving the cooler and making the entries.

Upon inspection of the cooler and custody seals, the sample custodian opens and inspects the contents of the cooler, and records the cooler temperature. If the cooler arrival temperature exceeds the required or method specified temperature range by $\pm 2^{o}C$ (for samples with a temperature requirement of $4^{o}C$, a cooler temperature of just above the water freezing temperature to $6^{o}C$ is acceptable); sample receipt is considered “compromised” and the procedure described in Section 4.7.1 is followed. All documents are immediately inspected to assure agreement between the test samples received and the COC.

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Any non-conformance, irregularity, or compromised sample receipt as described in Section 4.7.1 must be documented 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 permanent project record.

Samples that are being tested at another STL facility or by an external subcontractor shall be appropriately packaged, and sent out under COC.

Following sample labeling as described in Section 5.7.2, the sample is placed in storage. Sample storage is required to be access-controlled. All samples are stored according to the requirements outlined in the test method and in a manner such that they are not subject to cross contamination or contamination from their environment. Unless specified by method or state regulation, a tolerance range of $4 \pm 2^{o}C$ is used. Sample storage temperatures are monitored daily.

5.7.2. Sample Identification and Traceability

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 sample identification label.

All unused portions of samples, including empty sample containers, are returned to the secure sample control area.

5.7.3. Sub-sampling

Sample preparation procedures must be referenced in each STL facility's LQM and documented in the laboratory SOPs.

5.7.4. Sample Preparation

Sample preparation procedures must be referenced in each STL facility's LQM and documented in the laboratory SOPs.

5.7.5. Sample Disposal

Each facility shall have an SOP describing sample retention and disposal procedures. Samples should be retained in STL storage facilities 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 (example, 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Samples may be returned to the client per written request. Unused portions of samples found or suspected to be hazardous according to state or

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federal guidelines may be returned to the client upon completion of the analytical work.

Samples shall be disposed of in accordance with federal, state and local regulations. Each facility must have an SOP detailing the disposal of samples, digestates, and extracts. All laboratories shall remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated).

5.8. Assuring the Quality of Test Results

5.8.1. Proficiency Testing

Each STL facility must analyze Proficiency Test (PT) samples as required for accreditation. As required by NELAC, each STL facility participates in the PT program semi-annually for each PT Field of Testing (FoT) for which it is accredited, according to the NELAC PT FoT published guidelines. Under SDWA, the laboratory also analyzes a PT sample by each method once per year, if the laboratory uses more than one method for the analyte.

In addition to the PT program required for NELAC accreditation, STL participates in a number of additional PT programs, as appropriate for the specific facility.

PT samples must be handled and tested in the same manner (procedural, equipment, staff) as environmental samples. PT test sample data is archived using the requirements for project and raw data record retention.

Each STL facility performing chemical analyses also participates in a double blind proficiency test evaluation annually. An external vendor is contracted to submit double blind samples to the STL facility. Both the level of customer service and the accuracy of the test results are assessed objectively by the external contractor, who provides a detailed report to the Quality Director and to each of the STL facilities. This is administered as a double blind program in order to assess all facets of STL operations.

5.8.2. Control Samples

Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity, selectivity, and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch. Control samples must be uniquely identified and correlated to unique batches. There are also a number of QC sample types that monitor field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Control sample types and typical frequency of their application are outlined in Table 10. Note that frequency and use of control samples vary with specific regulatory, methodology and project specific criteria.

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Table 10 does not define STL's approach to the application of QC samples for each regulatory program or test method.

5.8.3. Calibration

Each STL Facility must define calibration protocols in STL facility SOPs.

5.8.4. Glassware Cleaning

5.8.5. Permitting Departures from Documented Procedure

Each STL facility must have a procedure that defines the process, documentation, and level of authorization required to permit departures from documented procedures.

Table 10 Control Samples

Laboratory QC Sample Type	Use	Required Frequency
Laboratory Control Sample (LCS) (Laboratory Fortified Blank)	Measures accuracy of the method in a blank matrix	1 per batch of 20 or less samples per matrix type per sample extraction or preparation method[1]
Method Blank (MB)	Measures method contribution to any source of contamination	1 per batch of 20 or less samples per matrix type per sample extraction or preparation method[1]
Instrument Blank	Measures instrumental contribution to any source of contamination	As specified in test method
Cleanup Blank	Measures clean up step contribution to any source of contamination	As specified in test method
Storage Blank	Measures storage contribution to any source of contamination (Volatiles only)	As specified in test method or SOP
Control, Brine Control, or Dilution Water	Measures the effect of blank water on test organisms (Aquatic toxicology)	As specified in test method and permit
Reference Toxicant	Measure sensitivity of test organisms (Aquatic toxicology)	Annually
Field QC Sample Type	**Use**	**Typical Frequency**
Matrix Duplicate	Measures the effect of the site matrix on the precision of the method	Per 20 samples per matrix or per SAP/QAPP[1,2]
Matrix Spike	Measures the effect of the site matrix on the accuracy of the method	Per 20 samples per matrix or per SAP/QAPP[1]
Matrix Spike Duplicate	Measures the effect of the site matrix on the precision of method	Per 20 samples per matrix or per SAP/QAPP[1,2]
Equipment Blank (Equipment Rinsate)	Measures field equipment contribution to any source of contamination	Per SAP/QAPP
Trip Blank	Measures shipping contribution to any source of contamination (Volatiles only)	Per Cooler
Field Blank	Measures the field environment contribution to any source of contamination	Per SAP/QAPP
Field Duplicate	Measures representativeness of the sampling and the effect of the site matrix on precision	Per SAP/QAPP

[1] Denotes an STL required frequency
[2] Either an MSD or an MD is required per 20 samples per matrix or per SAP/QAPP.

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Where a departure from a documented SOP, test method, or policy is determined to be necessary, or unavoidable, the departure shall be documented and be authorized by the appropriate level of management, which is defined in the policy. In some instances, it is appropriate to inform the client before permitting a departure. Any such occurrence is documented in the cover letter and/or project narrative.

5.8.6. Development of QC Criteria, Non-Specified in Method/Regulation

Where a method or regulation does not specify acceptance and/or rejection criteria, the laboratory must develop a policy for doing so. The policy must address how the laboratory examines the data user's needs and the demonstrated sensitivity, accuracy and precision of the available test methods in determining appropriate QC criteria.

Data users often need the laboratory's best possible sensitivity, accuracy, and precision using a routinely offered test method, or are unsure of their objectives for the data. For routine test methods that are offered as part of STL's standard services, the laboratory bases the QC criteria on statistical information such as determination of sensitivity, historical accuracy and precision data, and method verification data. The method SOP includes QC criteria for ongoing demonstration that the established criteria are met (e.g., acceptable LCS accuracy ranges, precision requirements, method blank requirements, initial and continuing calibration criteria, etc.).

In some cases, a routine test method may be far more stringent than a specific data user's needs for a project. The laboratory may either use the routinely offered test method, or may opt to develop an alternate test method based on the data user's objectives for sensitivity, accuracy, and precision. In this case, it can be appropriate to base the QC criteria on the data user's objectives, and demonstrate through method verification and ongoing QC samples that these objectives are met.

For example, a client may require that the laboratory test for a single analyte with specific DQOs for sensitivity, accuracy, and precision as follows: Reporting Limit of 10 ppm, accuracy $\pm$25%, and RSD of less than 30%. The laboratory may opt to develop a method that meets these criteria and document the results of the Method Blanks, MDL study, and LCSs that the method satisfies those objectives. In this case, both the method and the embedded QC criteria have been based on the client's DQOs.

In some cases, the data user needs more stringent sensitivity, accuracy, and/or precision than the laboratory can provide using a routine test method. In this case, it is appropriate that the laboratory provide documentation of the sensitivity, accuracy, and precision obtainable to the data user and let the data user determine whether to use the best available method offered by the laboratory, or determine whether method development or further research is required.

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5.9. Project Reports

5.9.1. General

All STL Project Reports that are generated under NELAC requirements must contain the content as described in Section 5.9.2. The criteria described in Section 5.9.3 and 5.9.4 applies to all Project Reports.

5.9.2. Project Report Content

- Title
- Laboratory Name, Address, Telephone Number, Contact Person
- Unique Laboratory Project Number
- Total Number of Pages (report must be paginated)
- Name and Address of Client
- Client Project Name (if applicable)
- Laboratory Sample Identification
- Client Sample Identification
- Matrix and/or Description of Sample
- Dates: Sample Receipt, Collection, Preparation and/or Analysis Date
- Definition of Data Qualifiers
- Reporting Units
- Test Method

The following are required where applicable to the specific test method or matrix:

- Solid Samples: Indicate Dry or Wet Weight
- Whole Effluent Toxicity: Statistical package used
- If holding time < 72 hours, Sample Collection, Preparation and/or Analysis Time
- Indication by flagging where results are reported below the quantitation limit.

5.9.3. Project Narrative

A Project Narrative and/or Cover Letter shall be included with each project report and at a minimum includes an explanation of any and all of the following occurrences:

- Non-conformances
- “Compromised” sample receipt (see Section 4.7.1)
- Method Deviations
- QC criteria failures

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Project Release

The Laboratory Director or his/her designee must authorize the release of the project report with a signature.

Where amendments to project reports are required after issue, these shall be in the form of a separate document and/or electronic data deliverable. The revised report is clearly identified as revised with the date of revision and the initials of the person making the revision. Specific pages of a project report may be revised using the above procedure with an accompanying cover letter indicating the page numbers of the project revised. The original version of the project report must be kept intact and the revisions and cover letter included in the project files. The authorized SOP deviations, non-conformances and QC failures must be covered in the case narrative, cover letter or within the report.

5.9.4. Subcontractor Test Results

Project reports from a subcontract laboratory shall not be altered, and shall be included in original form in the final project report provided by STL. Data from subcontractors' reports may be added to an STL electronic deliverable.

Subcontracted data shall be clearly identified as such, and the name and address of the laboratory performing the test shall be included in the project report. If the report is being generated under NELAC requirements, all information outlined in Section 5.9.2 are required for both the originating laboratory and the subcontracting laboratory.

Data subcontracted within STL may be reported on the originating laboratory's report forms provided the following mandatory requirements are met:

- The name, address, and telephone number of the facility are provided.
- Analytical results produced by the STL intra-company subcontractor are clearly identified as being produced by the subcontractor facility.
- The intra-company subcontractor's original report, including the COC, is retained by the originating laboratory.
- Proof of certification is retained by the originating laboratory.
- All information as outlined in Section 5.9.2 is included in the final report where the report is required to be compliant with NELAC, for both the originating and subcontracting laboratory.

5.9.5. Electronic Data Deliverables

Electronic Data Deliverables (EDD) are routinely offered as part of STL's services. STL offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), Format A, Excel, Dbase, GISKEY, and Text Files.

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EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process outlined in Section 4.4.1. Once the facility has committed to providing diskettes in a specific format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained as a QC record.

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.

5.9.6. Project Report Format

STL offers a wide range of project reporting formats, including EDDs, short report formats, and complete data deliverable packages modeled on the Contract Laboratory Protocol (CLP) guidelines. More information on the range of project reports available can be obtained by contacting any STL facility. Regardless of the level of reporting, all projects must undergo the levels of review as described in Section 5.3.6.

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Appendix: List of Quality System Policies and Procedures

QMP Citation	Description	Reference
1.2	Quality Policy	QMP
4.4	Contract Review	QMP
4.4.2	Project Planning Process	LAB Procedure
4.7.1	Sample Acceptance Policy	LAB Procedure
4.5	Subcontracting	QMP
5.3.2	Approved SOP Listing	LAB Procedure
4.3.2	Document Control	S-Q-001 & Lab Procedure
4.12.2	Record Retention & Purging	QMP
4.6	Purchasing Services and Supplies	QMP
4.7.2	Client Confidentiality	QMP
4.8	Complaints	QMP
4.9	Document and Control of Non-conformances	LAB Procedure
4.10	Corrective Action process	LAB Procedure
4.15.2	Quality Systems Management Review	QMP
4.11	Preventive Action Process	LAB Procedure
4.12.4	Archives and Record Transfer	QMP
4.13	Internal Audits	QMP
4.15	Management Reviews	QMP
5.1.2	Training	QMP
5.1.3	Ethics Policy	P-L-006
5.3.2	SOP Index	LAB Procedure
5.3.5	Method Detection Limit Studies	S-Q-003
5.3.5	Relationship of Limit of Detection to Quantitation Limit	LAB Procedure
5.3.7	Data Integrity and Security	QMP
5.3.6	Data Review	QMP
5.4.1	Equipment Operation	QMP
5.4.1	Equipment Tracking List	LAB Procedure
5.4.2	Equipment Maintenance	QMP
5.4.3	Equipment Verification and Calibration	QMP
5.4.3	Selection of Calibration Points	P-T-001
5.5	Measurement Traceability	QMP
5.5.1	Procedures for Checking Specifications for Ancillary Equipment	LAB Procedure
5.5.2	Reference Standards Traceability	QMP
5.7	Sample Handling, Transport and Storage	QMP
5.7.2	Sample Identification and Traceability	QMP
5.7.3	Subsampling	QMP
5.7.4	Sample Preparation	QMP
5.7.5	Sample Disposal	LAB Procedure
5.8.3	Calibration	LAB Procedure
5.8.4	Glassware Cleaning Procedures	LAB Procedure
5.8.5	Permitting Departures From Documented Procedures	LAB Procedure
5.8.6	Development of QC Criteria, Non-specified in Methods/Regulations	QMP
5.9	Reporting Analytical Results	QMP

Note: Where “QMP” is referenced it indicates the policy or procedure is covered by the QMP and not covered by a corporate procedure, and it does not require a laboratory specific procedure. However, when QMP is listed, the laboratories’ may still address it in more detail in their LQM or laboratory quality system procedures. When “LAB Procedure” is indicated, it requires the laboratory to address the item in its LQM or have a have a specific laboratory quality system policy or procedure for that item. Where a procedure number is listed, it refers to a corporate policy or procedure.

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STL

Implementation Date: 5/7/07

STL PITTSBURGH STANDARD OPERATING PROCEDURE

TITLE: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES, SW-846 METHOD 6010B AND EPA METHOD 200.7

(**SUPERSEDES:** PITT-MT-0001, Revision 8)

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1. SCOPE AND APPLICATION

1.1 This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Method 6010B and EPA Method 200.7. Table I of Appendix A lists the elements appropriate for analysis by Methods 6010B and 200.7. Additional elements may be analyzed under Methods 6010B and 200.7 provided that the method performance criteria presented in Section 13.0 are met.

1.2 ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used. For instance, in comparison to conventional ICP technique, ICP-Trace can achieve detection levels comparable to those determined using the graphite furnace atomic absorption spectroscopy (GFAAS) technique.

1.3 Method 6010B is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, tissues, wipes and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of dissolved samples and this must be clarified and documented before project initiation. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.4 Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met. Silver concentrations must be below 0.1 mg/L in aqueous samples and 50 mg/kg in solid matrix samples.

1.5 For DoD QSM Version 3 requirements, refer to SOP PITT-QA-DoD-0001.

2. SUMMARY OF METHOD

2.1 This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines 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. The position used must be free of spectral interferences and 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. The possibility of additional interferences should also be recognized and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

2.2 Refer to the appropriate SOPs for details on sample preparation methods.

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3. DEFINITIONS

3.1 Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2 Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3 Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4 Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4.0 INTERFERENCES

4.1 Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. 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.
- Stray light from the line emission of high concentration elements.

4.1.4 Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

4.1.1 A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2 Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3 Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

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5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.3 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.
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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5 The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.6 Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume

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hood, or under other means of mechanical ventilation. Metals digestates can be processed outside of a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.7 The preparation of standards and reagents will be conducted in a fume hood or well-ventilated area.

5.8 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1 Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2 Radio Frequency Generator.

6.3 Argon gas supply, welding grade or equivalent.

6.4 Coolflow or appropriate water cooling device.

6.5 Peristaltic Pump.

6.6 Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7 Class A volumetric flasks.

6.8 Autosampler tubes.

7. REAGENTS AND STANDARDS

7.1 Intermediate standards are purchased as custom STL multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met.

7.2 Working calibration and calibration verification solutions may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids. An exception to this is in the event the Trace ICP is utilized without the internal standard. In this case, the standard acid matrix must be matched to the final preparation matrix.

7.3 Refer to Tables III, IV, IVA, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.4 Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5 Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6 Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Sample holding times for metals are six months from time of collection to the time of analysis.

8.2 Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not

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required. Preservation must be verified prior to analysis.

8.3 Soil and wipe samples do not require preservation but must be stored at 4°C ± 2° until the time of preparation. Tissue samples are stored frozen until preparation.

9. QUALITY CONTROL

Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1 See Document QA-003 "STL Quality Control Program" for additional detail on criteria and corrective actions.

9.2 Initial Demonstration of Capability

Prior to analysis of any analyte using either Method 200.7 or Method 6010B, the following requirements must be met.

9.2.1 Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in STL QA Policy PITT-QA-007. The spike level must be between the calculated MDL and 10X the MDL to be considered valid. The result of the MDL determination must be below the STL reporting limit (RL). MDL studies for the determination of metals in soil need not be performed; an appropriate soil MDL may be computed from the experimentally determined MDL for metals in aqueous solution. Unless otherwise indicated by project or program requirements, we will report to the MDL as determined in 40 CFR Part 136, Appendix B.

9.2.2 Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL will be determined quarterly (every 3 months). If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples).

9.2.2.1 **DoD samples cannot be analyzed without a valid IDL.**

9.2.2.2 **For DoD, the established IDL must be less than the MDL for each analyte.**

9.2.3 Linear Range Verification (LR) - The linear range will be determined on a quarterly basis for each analyte wavelength used on each instrument. The standards used to define the linear range limit must be analyzed during a routine analytical run. For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard should be near the upper limit of the estimated range. The determined concentration of the linear range standards must be within 5% of the true value. The linear range is the concentration above which results cannot be reported without dilution of the sample. If the instrument is adjusted in any way that may affect the LR's, the LR's must be redetermined. The LR data must be documented and

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kept on file.

9.2.4 Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the facility-specific instrument operation SOP and ICP instrument manual for specific procedures to be used in setting background correction points.

9.2.5 Inter-element Corrections (IECs) - ICP interelement correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the facility specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than ± the RL as defined in Tables I, IA or II. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response.

9.2.6 Rinse Time Determination - Rinse times must be determined whenever a new instrument is set up. . To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see 9.2.4) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data. Linear Range Verifications are performed at a minimum of every six months. Whenever Linear Range Verifications are performed the suitability of the rinse time settings will be evaluated and the rinse time determination will be repeated when necessary.

9.3 Method Blank (MB) - One method blank must be processed with each preparation batch of up to 20

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samples. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level). **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the method blank.**

- If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples, which have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.4 Laboratory Control Sample (LCS) - One aqueous LCS (referred to as a Laboratory Fortified Blank in 200.7) must be processed with each preparation batch of up to 20 samples. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table III (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, for method 6010B, a control limit of 80 - 120% (85-115% for 200.7) recovery must be applied.

- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- In the instance where the LCS recovery is greater than 120% (115% for 200.7) and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the report narrative.**

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

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- For dissolved metals samples, which have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch of up to 20 samples (6010B) or one MS for every 10 or fewer samples (200.7). A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added (referred to as a Laboratory Fortified Matrix in 200.7). A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables III and VI (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For method 6010B, control limits of 75 - 125% (70 – 130% for 200.7) recovery and 20% RPD or historical acceptance criteria must be applied to the MS/MSD. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the MS.** If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples by 200.7, which have not been digested, a MS must be performed per every 10 or fewer samples by spiking an aliquot of the sample at the levels specified in Table III (Appendix A).

9.6 Dilution test – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5x (1:4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50x the MDL. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.7 Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate readings of four exposures of the ICV standard. For Method 6010B, the ICV must fall within 10% of the true

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value for that solution with relative standard deviation <5% from replicate (minimum of three) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the ICB.** If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. This standard is equivalent to the Quality Control Standard (QCS) and the first Instrument Performance Check (IPC) specified in 200.7.

9.8 Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV is a mid-range standard made from a dilution of the calibration standard. The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of three) exposures. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the CCB.** If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, reanalysis and recalibration are not required before continuation of the run. If a mid-run CCV or CCB fails, the analytical run may be continued; however, the result(s) for the affected element(s) may only be reported when bracketed by valid CCV/CCB pairs. If analytical results for one or more elements are not bracketed by valid CCV/CCB pairs, the problem must be corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed for those elements only.

9.9 Reporting Limit Verification Standard (RLV) or CRA – Calibration accuracy at the laboratory reporting limit is verified after the analysis of the ICB by running the RLV or CRA. This standard is at the reporting limit. Until in-house control limits are established, an in-house control limit of 50 – 150% recovery will be applied. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the RLV standard.**

9.10 Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom STL multielement ICS solutions must be used. All analytes should be spiked into the ICSAB solution therefore, if a non-routine analyte is required then it should be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display over correction as a negative number then the non-routine elements can be controlled from the ICSA as described in section 9.10.3. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.10.1 The ICSA and ICSAB solutions must be run at the beginning of the run. (See Section 11.11 for required run sequence.)

9.10.2 The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

9.10.3 ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the STL guidelines of ± 2x RL from zero. ICSA results for the non-interfering elements with RLs > 10 µg/L must fall within the STL guidelines of ± 1x RL from zero. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the ICSA.** If the ICSA results for the non-interfering elements do

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not fall within +/- 2x RL (RL ≤10) or ± 1xRL (RL>10) from zero the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
- If the affected element was not required then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration, which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.
- If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

9.11 Post-Digestion Spike Samples (PDS) - **For DoD samples, a post digestion spike will be run on a sample if the if the MS/MSD for the sample falls outside of % recovery criteria.** A post digestion spike is a matrix spike on a sample, which is added after the sample preparation is completed. The spike recovery from the post digestion spiked sample should be within the range 75-125% where the spike value is greater than 25% of the indigenous analyte concentration.

9.12 Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences, which cause a baseline shift. Refer to Section 11.14 for additional information on when MSA is required as well as Appendix D for specific MSA requirements.

9.13 Quality Assurance/Project Summaries - Certain clients may require project- or program-specific QC, which may supersede this SOP's requirements. Quality Assurance Summaries (QASs) or equivalent documents providing project-specific requirements should be developed so that project staff clearly understands the special project requirements.

10. CALIBRATION AND STANDARDIZATION

10.1 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.2 The instruments are profiled and calibrated according to the manufacturer's recommended procedures. Thermo has set up the ICP 61E to be profiled on Cu and the Trace ICPs are to be profiled on As. All other lines are preset by Thermo and should not be adjusted by the user. Flush the system with the calibration blank. The calibration curve must consist of a minimum of a blank

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and a standard. Refer to the facility-specific instrument SOP or ICP instrument manual for a detailed set up and operation protocols.

10.3 Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

10.4 Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions. The NELAC requirement for verification of the initial calibration at varied concentrations is met daily since the ICVs, CCVs, and RLVs/CRA are all at different concentrations.

11. PROCEDURE

11.1 For 200.7 analyses, dissolved (preserved) samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. Visibly transparent with a turbidity measurement of 1 NTU or less.

B. Is of one liquid phase and free of particulate or suspended matter following acidification.

C. Is NOT being analyzed for silver.

If the above criteria are met, the dissolved samples can be analyzed directly after an appropriate amount of 1:1 nitric acid is added to an aliquot of sample to adjust the acid concentration to approximately a 1% (v/v) nitric acid solution. Allowance for sample dilution should be made in the calculation.

11.2 A minimum of three exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.3 Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 9.2.6 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

11.4 The use of an autosampler for all runs is strongly recommended.

11.5 The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV, CCV, RLV/CRA), blanks (ICB, CCB, PB), interference checks (ICSA, ICSAB) and field samples (linear range) to improve the data review process.

11.6 To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.7 To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run. If any digestate for Method 200.7 has silver detected above 100 mg/L, add 1.0 ml of concentrated HCl to the digestate, mix and reanalyze. If the second analysis yields a higher value for silver, the second analysis is reported and discussed in the report narrative.

11.8 The use of an internal standard is recommended on the conventional, non-Trace ICPs as an alternative to using the method of standard additions. This technique is useful in overcoming matrix interferences especially in high solids matrices. However, for conventional ICP techniques, internal standards may not be necessary provided that one of the following is performed to minimize physical interferences: (1)

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peristaltic pump is used, (2) high solids nebulizer is used, or (3) high solids samples are diluted and reanalyzed.

11.9 The use of an internal standard is **required** on the Trace ICP unless the calibration and QC standards are matrix matched to each digestion procedure. The following procedural guidelines must be followed when using an internal standard:

11.9.1 Typically used internal standards are: yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.9.2 The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.9.3 The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.9.4 The internal standard raw intensity counts must be printed on the raw data.

11.9.5 The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

11.9.5.1 If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

11.9.5.2 If the internal standard counts in the field samples are more than ±30% higher than the expected level, the field samples must then be:

(1) Diluted and reanalyzed;

(2) The IS concentrations must be raised; or

(3) A different internal standard must be used.

11.10 The following analytical sequence must be used for Methods 6010B and 200.7:

Instrument Calibration
ICV
ICB
RLV/CRA
ICSA
ICSAB
7 samples
CCV
CCB
10 samples
CCV

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CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB
Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010B and 200.7 quality control criteria.

11.11 Full method required QC must be available for each wavelength used in determining reported analyte results.

11.12 Guidelines are provided in the appendices on procedures to minimize contamination of samples and **standards, preventive maintenance and troubleshooting.**

11.13 All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data are established. If an interelement correction exists for an analyte, which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.14 For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

- recovery of the analyte in the matrix spike is not at least 50%,
- the concentration of the analyte does not exceed the regulatory level, and,
- the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix D provides guidance on performing MSA analyses.

11.15 Any variation in procedure shall be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report and is approved by a Supervisor/Group Leader and QA Manager. If contractually required, the client shall be notified by the Project Manager.

11.16 Nonconformance documentation shall be filed in the project file.

11.17 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1 ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2 CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

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12.3 RLV/CRA percent recoveries are calculated using the same equation as the ICV or CCV (replace ICV or CCV with RLV/CRA in the above equations).

12.4 Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.5 The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

12.6 The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{CxV1xD}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.7 The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg/Kg, dryweight = \frac{CxVxD}{WxS}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight

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concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.8 The final concentration determined in digested wipe samples is calculated as follows:

$$ug/wipe = (C \times V \times D \times 1000)$$

Where:
C = Concentration (mg/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor

12.9 The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.10 The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} x100$$

Where:
I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading $\times$ 5)

12.11 Appropriate factors must be applied to sample values if dilutions are performed.

12.12 Sample results should be reported with up to three significant figures in accordance with the STL significant figure policy.

13. METHOD PERFORMANCE

13.1 Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2 Refer to Tables I, IA & II in Appendix A for the list of Method 6010B and 200.7 analytes as well as additional analytes that may be analyzed using this SOP.

13.3 Method performance is determined by the analysis of MS and MSD samples as well as method blanks and laboratory control samples. The MS or MSD recovery should fall within +/- 25% (6010B) or +/- 30% (200.7) and the MS/MSD should compare within 20% RPD or within the laboratory's historical acceptance limits. These criteria apply to analyte concentrations greater than or equal to 10xIDL. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the MS.** Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% (15% for 200.7) of the true value or within the laboratory's historical acceptance limits. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the method blank.**

13.4 Training Qualification:

The group/team leader or the supervisor has the responsibility to ensure that this procedure is

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performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1 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 This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1 15.1 The following waste streams are produced when this method is carried out.

15.1.1 Acid waste consisting of sample and rinse solution This waste is collected in waste containers identified as "Acid Waste", Waste #33.

15.1.2 Expired Metals Standards – This waste is collected in containers identified as "Acid Waste with Metals", Waste #6.

16. REFERENCES

16.1 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits.

16.2 Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

16.3 Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7.

16.4 Standard Methods 20th Edition 2340B; Hardness by Calculation

16.5 QA-003, STL Pittsburgh QC Program.

16.6 QA-004, Rounding and Significant Figures.

16.7 PITT-QA-007, Method Detection Limits.

16.8 PITT-QA-DoD-0001, Implementation of the DoD QSM Version 3.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1 Modifications/Interpretations from reference method

17.1.1 Modifications/interpretations from both Methods 6010B and 200.7.

17.1.1.1 STL laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.

17.1.1.2 Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. In determining IECs, because of lack of definition clarification for "concentration range around the calibration blank," STL has adopted the procedure in EPA CLP ILM04.1.

17.1.1.3 Section 8.5 of Method 6010B and Section 9.5 of Method 200.7 recommend that whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because STL laboratories receive no prior information from clients regarding when to expect a new

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or unusual matrix, STL may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At STL labs, matrix interference is determined by evaluating data for the LCS and MS/MSD. STL requires documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.1.2 Modifications from Method 200.7.

17.1.2.1 The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.2 Section 7.12 of 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations, which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.3 The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 section 10.4 states that results should fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. STL Pittsburgh follows the CLP ICS procedures because it is applicable to a wider number of programs. Therefore, we feel it is a more conservative approach.

17.1.2.4 Method 200.7 section 9.3.4 states the CCB should be less than the IDL, but > the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. STL has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. SOP section 9.8 provides the detailed corrective action criteria that must be followed. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the CCB.**

17.1.3 Modifications from Method 6010B.

17.1.3.1 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the method blank.**

17.1.3.2 Method 6010B section 8.6.1.3 states that the results of the calibration blank are to agree within 3x the IDL. If not, repeat the analysis two or more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. STL has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. See SOP Section 9.8 for a detailed description of the required corrective action procedures. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the calibration blanks.**

17.2 Documentation and Record Management

- The following documentation comprises a complete ICP raw data package:
- Raw data (direct instrument printout).
- Relevant sample preparation benchsheets.
- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).

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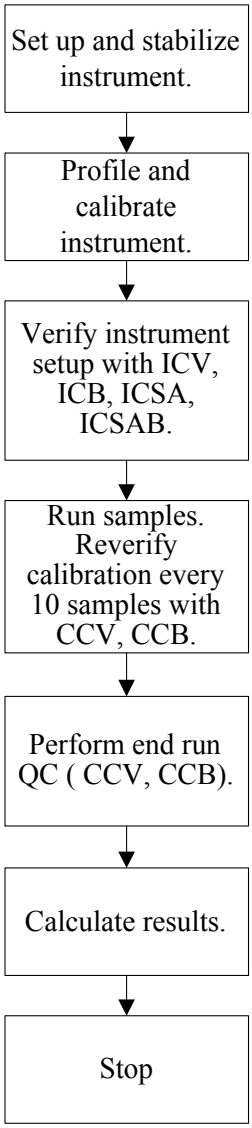
- Data review checklist - See Appendix B.
- Standards documentation (including prep and expiration dates, source, and lot #).
- Nonconformance/anomaly documentation (if applicable).

17.3 Flow Diagram



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APPENDIX A

TABLES

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TABLE I. Method 200.7 and 6010B Target Analyte List

ELEMENT	Symbol	CAS #	6010B analyte	200.7 analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil	Reporting Limit (ug/wipe) Wipe
Aluminum	Al	7429-90-5	X	X	200	20	10
Antimony	Sb	7440-36-0	X	X	60	6	3
Arsenic	As	7440-38-2	X	X	300	30	15
Barium	Ba	7440-39-3	X	X	200	20	10
Beryllium	Be	7440-41-7	X	X	4.0	0.4	0.25
Boron	B	7440-42-8	X	X	200	20	10
Cadmium	Cd	7440-43-9	X	X	5.0	0.5	0.25
Calcium	Ca	7440-70-2	X	X	5000	500	250
Chromium	Cr	7440-47-3	X	X	10	1	0.5
Cobalt	Co	7440-48-4	X	X	50	5	2.5
Copper	Cu	7440-50-8	X	X	25	2.5	1.25
Iron	Fe	7439-89-6	X	X	100	10	5
Lead	Pb	7439-92-1	X	X	100	10	5
Lithium	Li	7439-93-2	X	X	50	5	2.5
Magnesium	Mg	7439-95-4	X	X	5000	500	250
Manganese	Mn	7439-96-5	X	X	15	1.5	0.75
Molybdenum	Mo	7439-98-7	X	X	40	4	2
Nickel	Ni	7440-02-0	X	X	40	4	2
Phosphorus	P	7723-14-0	X	X	300	30	NA
Potassium	K	7440-09-7	X	X	5000	500	250
Selenium	Se	7782-49-2	X	X	250	25	12.5
Silicon	Si	7631-86-9	X	X	500	N/A	N/A
Silver	Ag	7440-22-4	X	X	10	1	0.5
Sodium	Na	7440-23-5	X	X	5000	500	250
Strontium	Sr	7440-24-6	X	X	50	5	2.5
Thallium	Tl	7440-28-0	X	X	2000	200	100
Vanadium	V	7440-62-2	X	X	50	5	2.5
Zinc	Zn	7440-66-6	X	X	20	2	1

Note: Where reporting “Hardness” by ICP use the following equations per SM20th ed. 2340B:

Calcium Hardness = 2.497 [Ca, mg/L]

Total Hardness = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]

Where reporting “Silica” by ICP use the following equation:

Silica = Silicon * 2.14

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TABLE IA. Method 200.7 and 6010B Trace ICP Target Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil	Reporting Limit (ug/wipe) Wipe
Arsenic	As	7440-38-2	10	1.0	0.5
Lead	Pb	7439-92-1	3.0	0.3	0.15
Selenium	Se	7782-49-2	5.0	0.5	0.25
Thallium	Tl	7440-28-0	10	1.0	0.5
Antimony	Sb	7440-36-0	10	1.0	0.5
Cadmium	Cd	7440-43-9	5.0	0.5	0.25
Silver	Ag	7440-22-4	5.0	0.5	0.25
Chromium	Cr	7440-47-3	5.0	0.5	0.25

TABLE II. Non-Routine Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil	Reporting Limit (ug/wipe) Wipe
Tin	Sn	7440-31-5	100	10	5
Titanium	Ti	7440-32-6	50	5	2.5

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TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Aluminum	2000	2000
Antimony	500	500
Arsenic	2000	2000
Barium	2000	2000
Beryllium	50	50
Cadmium	50	50
Calcium	50000	50000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1000	1000
Lead	500	500
Lithium	1000	1000
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Strontium	1000	1000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500
Boron	1000	1000
Silicon	10000	10000
Tin	2000	2000
Titanium	1000	1000

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TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	25000	50000
Antimony	10000	60	1000	5000
Arsenic	10000	300	1000	5000
Barium	10000	200	1000	5000
Beryllium	10000	4	1000	5000
Cadmium	10000	5	1000	5000
Calcium	100000	5000	25000	50000
Chromium	10000	10	1000	5000
Cobalt	10000	50	1000	5000
Copper	10000	25	1000	5000
Iron	100000	100	25000	50000
Lead	10000	100	1000	5000
Lithium	10000	50	1000	5000
Magnesium	100000	5000	25000	50000
Manganese	10000	15	1000	5000
Molybdenum	10000	40	1000	5000
Nickel	10000	40	1000	5000
Potassium	100000	5000	25000	50000
Selenium	10000	250	1000	5000
Silver	2000	10	500	1000
Sodium	100000	5000	25000	50000
Strontium	10000	50	1000	5000
Thallium	20000	2000	5000	10000
Vanadium	10000	50	1000	5000
Zinc	10000	20	1000	5000
Boron	10000	200	1000	5000
Silicon	10000	500	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000

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TABLE IVA. Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	12500	25000
Antimony	1000	10	250	500
Arsenic	1000	10	250	500
Barium	4000	10	1000	2000
Beryllium	4000	4	1000	2000
Cadmium	1000	5	250	500
Calcium	100000	5000	25000	50000
Chromium	4000	5	1000	2000
Cobalt	4000	50	1000	2000
Copper	4000	25	1000	2000
Iron	50000	100	12500	25000
Lead	1000	3	250	500
Magnesium	100000	5000	25000	50000
Manganese	4000	15	1000	2000
Molybdenum	4000	40	1000	2000
Nickel	4000	40	1000	2000
Potassium	250000	5000	50000	125000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	250000	5000	50000	125000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000
Boron	4000	200	1000	2000
Silicon	4000	500	1000	2000
Tin	4000	100	1000	2000
Titanium	4000	50	1000	2000

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TABLE V. Interference Check Sample Concentrations*

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	10000**
Vanadium	-	500
Zinc	-	1000
Tin	-	1000

* Custom STL solutions contain common analytes. Non-routine elements not listed above should be spiked into the ICSAB at 1000 ug/L.

** Thallium level for Trace ICP should be at 1000 ug/L.

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TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

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TABLE VII. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met	RSD between duplicate exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. Method 6010B: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero. (1)	Terminate analysis; Correct the problem; Recalibrate.
RLV/CRA	Beginning of every analytical run, immediately following the ICB.	50 – 150% recovery. (1)	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7 & 6010B: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero. (1)	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.10.3 (1)	See Section 9.10.3.
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.10.2.

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TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Dilution Test	One per prep batch.	For samples > 50x MDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. (1) Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.3). Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.3).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.3 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. (85-115% for 200.7) Samples for which the contaminant is < RL and the LCS results are > 120% (115% for 200.7) may not require redigestion or reanalysis (see Section 9.4)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.

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TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix Spike	One per sample preparation batch of up to 20 samples (6010B) or one per every 10 or fewer samples (200.7).	75 - 125 % (6010B) or 70 – 130% (200.7) recovery or in-house control limits. [1] For TCLP See Section 11.14.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 11.14.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20%. [1]	See Corrective Action for Matrix Spike.

[1] For specific DoD requirements, refer to PITT-QA-DoD-0001.

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APPENDIX B

STL ICP DATA REVIEW CHECKLIST

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STL – Pittsburgh Data Review Checklist - ICP

Run Date: _____ Lots Analyzed: 4. _____ 8. _____ 12. _____
Analyst: _____ 1. _____ 5. _____ 9. _____ 13. _____
Instrument: _____ 2. _____ 6. _____ 10. _____ 14. _____
Methods: _____ 3. _____ 7. _____ 11. _____ 15. _____

Review Item	Yes (□)	No (□)	N/A (□)	2nd Lv (□)	Comments
A. Calibration/Instrument Run QC 1. Instrument calibrated per manufacturer's instructions and at SOP specified levels?					
2. ICV/CCV analyzed at appropriate frequency and within control limits? (6010B, CLP=90-110%, 200.7=95-105%[ICV])?					
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?					
4. CRA/RLV/CRI analyzed? (CRI for CLP only)					
5. ICSA/ICSAB run at required frequency and within SOP limits?					
B. Sample Results 1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed?					
2. All reported results bracketed by in control QC?					
3. Sample analyses done within holding time?					
C. Preparation/Matrix QC 1. LCS done per prep batch and within QC limits?					
2. Method blank done per prep batch and < RL or CRDL (CLP)?					
3. MS run at required frequency and within limits?					
4. MSD or DU run at required frequency and RPD within SOP limits?					
5. Dilution Test done per prep batch (or per SDG for CLP)?					
6. Post digestion spike analyzed if required (CLP only)?					
D. Other 1. Are all nonconformances documented appropriately?					
2. Current IDL/LR/IEC data on file?					
3. Calculations checked for error?					
4. Transcriptions checked for error?					
5. All client/project specific requirements met?					
6. Date/Time of analysis verified as correct?					

General Comments: _____

Analyst & Date: _____ Second-Level Review & Date: _____

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APPENDIX C

CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 200.7, AND BY STL

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CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW6010B, AND STL INC. SOP

EPA 200.7	SW6010B	STL Inc. SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	n/a	n/a
Laboratory fortified blank (LFB)	n/a	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

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APPENDIX D

MSA GUIDANCE

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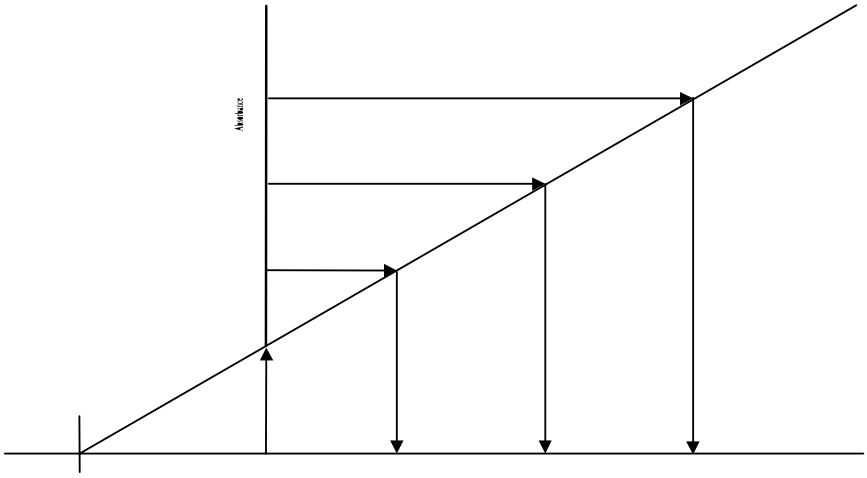
Appendix D. MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

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APPENDIX E

TROUBLESHOOTING GUIDE

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Problem	Possible Cause/Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Cu/Mn Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged) Realign torch Check IECs
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

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APPENDIX F

CONTAMINATION CONTROL GUIDELINES

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APPENDIX F. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 20% nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipette tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

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APPENDIX G

PREVENTIVE MAINTENANCE

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APPENDIX G. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily	Change sample pump tubing and pump windings Check argon gas supply level Check rinse solution and fill if needed Check waste containers and empty if needed Check sample capillary tubing is clean and in good condition Check droplet size to verify nebulizer is not clogged. Check sample flow for cross flow nebulizer Check Cu/Mn ratio-should be 30% of value at date that IECs were performed Check pressure for vacuum systems
As Needed	Clean plasma torch assembly to remove accumulated deposits Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance Replace peristaltic pump tubing, sample capillary tubing, and autosampler sipper probe
Weekly	Apply silicon spray on autosampler tracks Check water level in cool flow
Monthly	Clean air filters on back of power unit to remove dust Check D mirror for air instruments
Bi-yearly	Change oil for vacuum systems Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

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STL

Implementation Date: 5/7/07

STL PITTSBURGH STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY COLD VAPOR ATOMIC ABSORPTION, SW846 7470A AND MCAWW 245.1

(SUPERSEDES: PITT-MT-0005, REVISION 6)

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Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A and MCAWW Method 245.1. Both the water bath digestion and the autoclave digestion are available at the STL Pittsburgh facility, however the default practice is the autoclave digestion for 7470A. The water bath procedure is always used for 245.1. Both procedures are described in this SOP.

1.2. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.3. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, wipes, TCLP, EP and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed, however Method 7471A (see C-MT-0007) is usually the method of choice. All matrices require sample preparation prior to analysis.

1.4. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters, domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.5. The STL reporting limit for mercury in aqueous matrices is 0.0002 mg/L.

1.6. For DoD QSM Version 3 requirements, refer to SOP PITT-QA-DoD-0001.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride 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. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

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3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Potassium permanganate which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. Seawaters, brines and industrial effluents 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 at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample head space before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined

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by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6. The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.4. 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.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 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/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.
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.

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Potassium Permanganate	Oxidizer	5 Mg/M3 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.
Stannous Chloride	Irritant	2 Mg/M3 TWA as Tin	Causes irritation to the respiratory tract. Can irritate skin and eyes. Symptoms include coughing and shortness of breath. Contact with skin and/or eyes may cause redness, itching and pain.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.6. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since

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mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.6.1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4, or

5.6.2. Iodine, 0.25%, in a 3% KI solution.

5.7. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.8. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.9. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor or EH&S coordinator.

5.10. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.11. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath (capable of maintaining a temperature of 90-95 °C) or autoclave that is able to obtain conditions of 15 lbs., 120 °C for 15 minutes.

6.2. LEEMAN Labs PS200II Mercury Analyzer:

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6.2.1. LEEMAN Mercury Lamp P.N. 317-00048.

6.2.2. Peristaltic Pump.

6.2.3. Flow Meter.

6.2.4. Printer.

6.2.5. Dehydrator tube.

6.3. Leeman HYDRA AA Automated Mercury Analysis System.

6.4. Disposable Sealable Sample Containers (Corning).

6.5. Argon gas supply (ultrahigh purity-grade).

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Thermometer (capable of accurate readings at 95 °C).

6.9. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (1000 ppm) mercury standards (in 10% HNO_3) are purchased as custom STL solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Intermediate mercury standard (10 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The intermediate standard must be made monthly and must be prepared in a matrix of 2% HNO_3. This acid (2 mL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

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7.4. Working mercury standard (0.1 ppm): Take 1 mL of the intermediate mercury standard (7.3) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot. A second source working standard is prepared at 0.1 ppm for preparation of the ICV.

7.5. The calibration standards listed in Table I must be prepared fresh daily from the working standard (7.4) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into 100 mL flasks and diluting to volume with reagent water. The 0, .5, 1.0, 5.0 and 10 standards are recommended by Thermo Electron. The 0.2 standard level was selected to include a standard at the RL. See Table 1 (Appendix A) for the preparation of the ICV, CCV and RLV standards.

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.6. The initial calibration verification standard (ICV) must be made from a different stock solution than that of the calibration standards.

7.7. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.9. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9.1. Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated H_2SO_4 to 1 liter with reagent water.

7.10. Stannous chloride solution: Add 200 g of stannous chloride to 2 L of 10% hydrochloric acid.

7.11. Stannous sulfate may be used in place of stannous chloride. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

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7.12. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.13. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

7.14. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of analysis. For TCLP leachates, the holding time is 28 days from the time of TCLP extraction to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7470A or the 245.1, the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated

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sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate for 7470A or a matrix spike (one per 10 or fewer samples) for 245.1. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS, MSD) are not included in the sample count for determining the size of a preparation batch.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit, or above 10% of either the measured concentration of that analyte in associated samples or the regulatory limit. See QA-003 for more detail on criteria and corrective actions. In addition, blank contamination should always be evaluated against project specific requirements. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the method blank.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the project narrative.

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative and the client must be notified.

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9.5. Laboratory Control Sample (LCS) - One aqueous LCS (referred to as a Laboratory Fortified Blank in 245.1) must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. The CCV results can be reported as LCS results since all CCVs (as well as all other standards) are processed through the sample preparation step with the field samples. No more than 20 samples can be associated with one CCV used for the purpose of reporting LCS data.

- If the LCS is outside established control limits the system is out of control and corrective action must occur. Corrective action will result in the batch being re-prepped and re-analyzed. In-house control limits are 80 - 120% for SW-846 method 7470A and 85 – 115% for EPA method 245.1).

- In the instance where the LCS recovery is > 120% (7470A) or > 115% (245.1) and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the case narrative.

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch of up to 20 samples for 7470A or a MS must be processed for every 10 or fewer samples for 245.1. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added (referred to as a Laboratory Fortified Matrix in 245.1). A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are

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established, a control limit of 75 - 125 % (7470A) or 70 – 130% (245.1) recovery and 20% RPD must be applied to the MS/MSD. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the MS/MSD.** If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% (7470A) or 5% (245.1) of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the ICB.** If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the associated samples. The ICV is equivalent to the Quality Control Sample (QCS) and the first Initial Performance Check (IPC) specified in 245.1.

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the analytical sequence. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% (7470A) or 10% (245.1) of the true value for that solution. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the CCB.** Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples

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reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.9. Reporting Limit Verification Standard (RLV) – Calibration accuracy at the laboratory reporting limit is verified after the analysis of the ICB. Until in-house control limits are established, a control limit of 50 – 150% recovery will be applied.

9.10. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.2.13 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to CVAA instrument manual for detailed setup and operation protocols.

10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the STL reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

10.6. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

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10.7. Refer to Section 9.0 and Table II for calibration verification procedures, acceptance criteria and corrective actions. The NELAC requirement for verification of the initial calibration at varied concentrations is met daily since the ICVs, CCVs, and RLVs are all at different concentrations.

11. PROCEDURE

11.1. Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB, RLV) are processed through the digestion procedure as well as the field samples. *An exception to this is for Method 245.1 samples. The calibration curve samples are* ***not*** *heated.*

11.1.2. Transfer 100 mL of well-mixed sample or standard to a clean sample digestion bottle. Refer to PITT-QA-0024 for subsampling procedures.

Note: Reduced sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the same sample to reagent ratio. All samples and standards must be processed similarly.

11.1.3. Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 mixing after each addition.

Note: All spiking (LCS, MS, MSD) should be done after the initial addition of acids (see Appendix A, Table 1).

11.1.4. Add 15 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If after the addition of up to 25 mL additional permanganate the color does not persist, sample dilution prior to reanalysis may be required.

Note: When performing analyses using automated vs. manual techniques the sample dilution resultant from the addition of more than the original aliquot of permanganate solution must be compensated for by the addition of the same volume of permanganate to all associated samples, standards, *and QC samples (e.g. LCS and blank)* in the run. In instances, where this is not feasible, the addition of excess reagent can be addressed through mathematical correction of the results to account for the resultant dilution effect.

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11.1.5. Add 8 mL of potassium persulfate solution and heat for two hours in a water bath at 90 - 95 °C.

NOTE: Alternatively, for analyses using 7470A, samples may be digested using an autoclave for 15 minutes at 120 °C and 15 lbs (default).

11.1.6. Cool samples.

11.2. Sample Analysis:

11.2.1. Refer to the SOP PITT-MT-0028 and the instrument manuals for detailed setup and operation protocols for the LEEMAN PS200II and Hydra AA.

11.2.2. Refer to CVAA instrument manual for detailed setup and operation protocols.

11.2.3. When ready to begin analysis, add 6 mL of sodium chloride-hydroxylamine hydrochloride “clearing solution” to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains). Add this solution in 6 mL increments until the permanganate is completely reduced i.e. colorless.

11.2.4. Automated determination: Follow instructions provided by instrument manufacturer.

11.2.5. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. concentration of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.6. All measurements must fall within the defined calibration range to be valid. When sample concentrations exceed the upper limit of the calibration curve, the samples will be diluted and reanalyzed (if possible) to bring them within calibration curve. When reported sample concentrations either exceed the upper limit of the curve (i.e. cannot be rerun) or fall below the reporting limit, the data will be qualified as estimated. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.7. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

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11.2.8. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.9. The following analytical sequence must be used with 7470A and 245.1:

Instrument Calibration
ICV
ICB
RLV
Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Methods 7470A and 245.1.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.2.10. The following run sequence is consistent with 7470A, CLP and 245.1 and may be used as an alternate to the sequence in 11.2.11. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration
ICV
ICB
RLV or CRA*
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.
CCV
CCB

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Refer to the appropriate CLP SOPs (PITT-MT-0006) for quality control requirements for QC samples.

* Refer to the CLP SOPs for information on the CRA.

11.2.11. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and matrix spike levels for TCLP analyses are detailed in Table I (Appendix A). Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and parts maintenance. For instrument troubleshooting, use the auto diagnostics software. If the problem cannot be determined using the software, place a call to service personnel.

11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

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12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. RLV percent recoveries are calculated using the same equation as the ICV or CCV (replace ICV or CCV with RLV in the above equations).

12.4. Matrix spike recoveries are calculated according to the following equation:

$$\%\ R\ = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.5. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

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$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.6. The final concentration for an aqueous sample is calculated as follows:

$$mg/L = \text{C x D}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Appropriate factors must be applied to sample values if dilutions are performed.

12.9. Sample results should be reported with up to three significant figures in accordance with the STL significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Method performance is determined by the analysis of method blanks, laboratory control samples, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 25 % (7470A) or +/- 30% (245.1) and the matrix spike duplicates should compare within 20% RPD. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the MS.** The method blanks must meet the criteria in Section 9.4. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the method blank.** The laboratory control sample should recover within 20% (7470A) or 15% (245.1) of the true value until in house limits are established.

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13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. 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. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

15.1. The following waste streams are produced when this method is carried out.

15.1.1. Extracted sample containing less than 1 ppb Hg. This waste is collected in waste containers identified as "Acid Waste", Waste #33. It is neutralized to a pH between 6 and 9 and is disposed down a lab sink.

15.1.2. Unused Standards. This waste collected in containers identified as "Mercury Standards Waste", Waste #4.

15.1.3. Extracted sample containing greater than 1 ppb Hg. This waste collected in containers identified as "Mercury Standards Waste", Waste #4.

15.1.4. Mercury Analyzer Waste. Waste discharged from mercury analyzer is collected in containers identified as "Mercury Standards Waste", Waste #4.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

16.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, 1994, Method 245.1, Revision 3.0.

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16.3. U.S.EPA Statement of Work for Inorganics Analysis, ILM04.1.

16.4. QA-003, STL QC Program.

16.5. QA-004, Rounding and Significant Figures.

16.6. PITT-QA-007, Method Detection Limits.

16.7. PITT-QA-DoD-0001, Implementation of the DoD QSM Version 3.

16.8. PITT-QA-0024, Subsampling.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from both 7470A and 245.1.

17.1.1.1. The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2. This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology."

17.1.1.3. The alternate run sequence presented in Section 11.2.12 is consistent with method requirements.

17.1.2. Modifications from Method 7470A

17.1.2.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the

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reporting limit. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the method blank.**

17.1.2.2. Documentation is on file from EPA's Office of Solid Waste (Oliver Fordham 11/28/95) regarding the acceptance of the autoclave as an equivalent heating device to the water bath. In his letter, Mr. Fordham cited the CLP water protocol 245.1 CLP-M and therefore the operating parameters from that method were adopted for 7470A (15 minutes at 120 °C and 15 lbs.).

17.1.3. Modifications from 245.1

17.1.3.1. Method 245.1, Section 9.3 states concentrations should be reported as follows: Between 1 and 10 ug/L, one decimal; above 10 ug/L, to the nearest whole number. STL reports all Hg results under this SOP to two significant figures.

17.2. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Non-conformance summary (if applicable).

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Figure 1. Aqueous Sample Preparation - Mercury

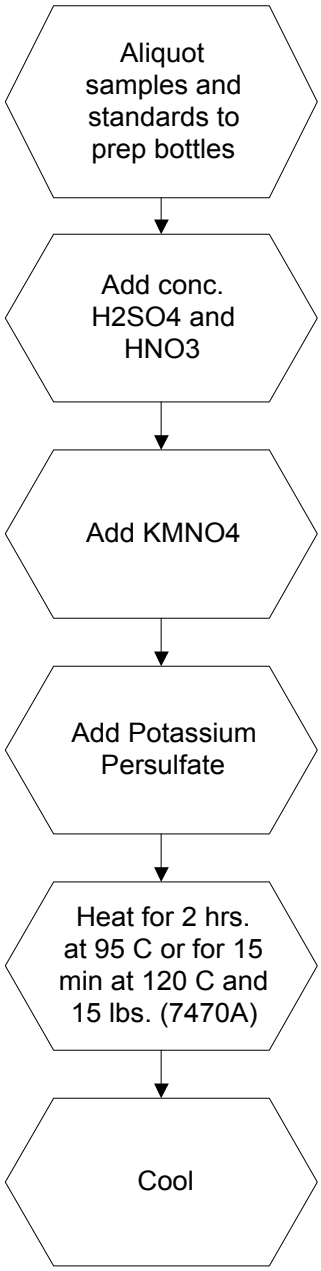

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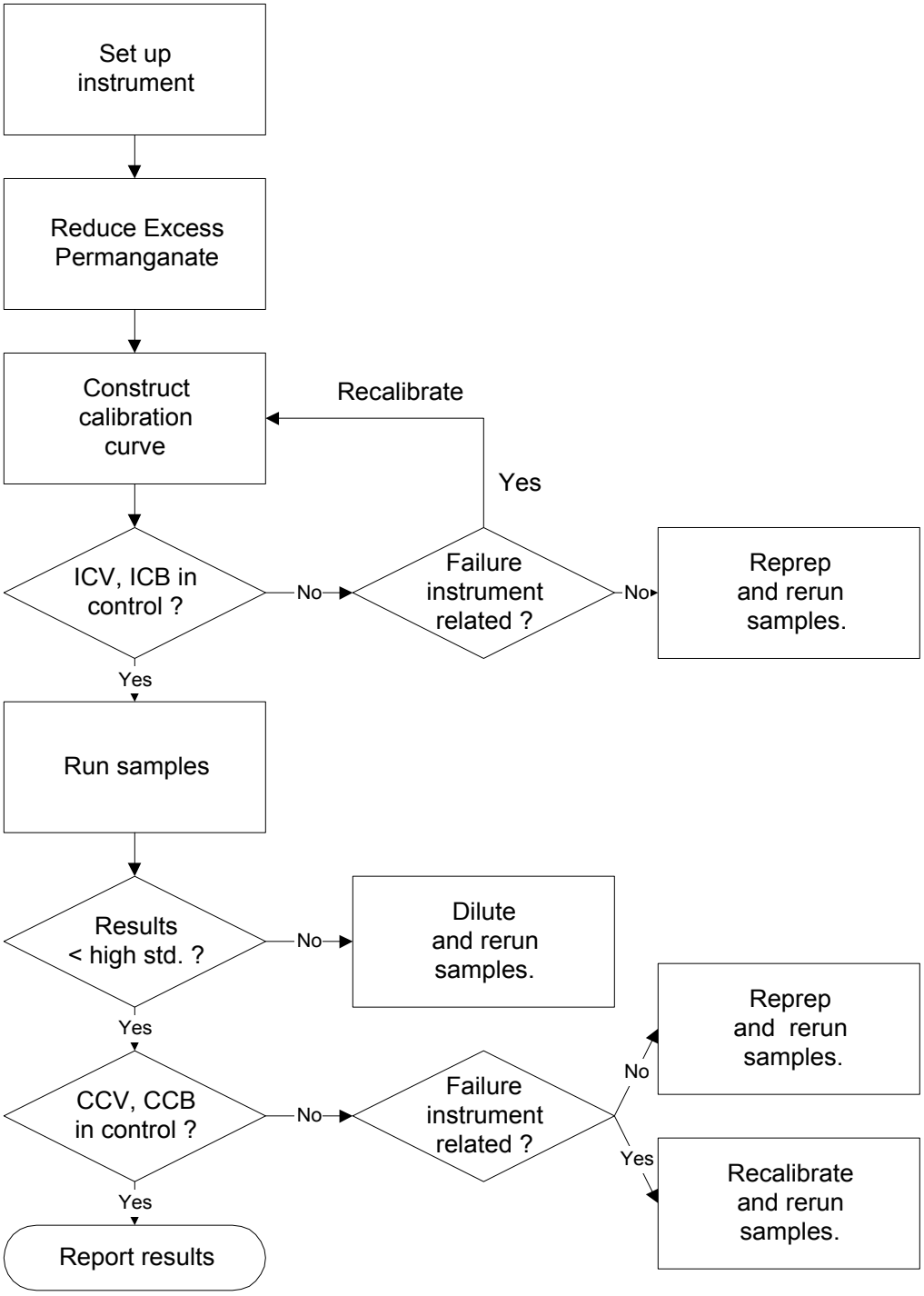
Figure 2. CVAA Mercury Analysis



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APPENDIX A

TABLES

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TABLE I . MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS (MG/L)

Method	Reporting Limit	
SW846 7470A	0.0002 mg/L	
SW846 7470A (TCLP)	0.0002 mg/L	
MCAWW 245.1	0.0002 mg/L	
Standard or QC sample	**mLs of 0.1 ppm Working Standard**	**Concentration (mg/L) *** **
Std 0	0	0
Std 1	0.2	0.0002
Std 2	0.5	0.0005
Std 3	1.0	0.001
Std 4	5.0	0.005
Std 5	10.0	0.010
ICV	2.5 **	0.0025
CCV	5.0	0.005
RLV	0.2	0.0002
LCS	2.5	0.0025
Aqueous MS	1.0	0.001
TCLP MS	5.0	0.005

*** SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements.**

**** Prepared from a second source 0.1 ppm working standard.**

***** When brought to a 100 mL final volume.**

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TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	7470A: 90 - 110 %. 245.1: 95 – 105%	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero. (1)	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
RLV	Beginning of every analytical run, immediately following the ICB.	50 – 150% recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.9).
CCV	Every 10 samples and at the end of the run.	7470A: 80 - 120 %. 245.1: 90 – 110%	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.8).
CCB	Immediately following each CCV.	The result must be within +/- RL from zero. (1)	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep batch (see Section 9.8).

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TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. (1) Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.4).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% (7470A) or 85 – 115% (245.1) recovery or in-house control limits.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples (7470A) or one for every 10 or fewer samples (245.1).	75 - 125 % (7470A) or 70 – 130% (245.1) recovery or in-house control limits. (1) If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.6) For TCLP see Section 11.2.13
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % (7470A) or 70 – 130% (245.1) recovery or in-house control limits; RPD ≤ 20%. (1) (See MS)	See Corrective Action for Matrix Spike.

(1) For specific DoD requirements, refer to PITT-QA-DoD-0001.

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APPENDIX B
STL Hg DATA REVIEW CHECKLIST

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STL Pittsburgh Data Review Checklist – Mercury

Run Date: _____ Lots Analyzed: 4. _____ 8. _____ 12. _____

Analyst: _____ 1. _____ 5. _____ 9. _____ 13. _____

Instrument: _____ 2. _____ 6. _____ 10. _____ 14. _____

Methods: _____ 3. _____ 7. _____ 11. _____ 15. _____

Review Item	Yes (✔)	No (✔)	N/A (✔)	2nd Level Review (✔)	Comments
A. Calibration/Instrument Run QC					
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels?					
2. ICV/CCV analyzed at appropriate frequency and within control limits?					
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?					
4. CRA run? (CLP only)					
B. Sample Results					
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?					
2. All reported results bracketed by in control QC?					
3. Sample analyses done within holding time?					
C. Preparation/Matrix QC					
1. LCS done per prep batch and within QC limits?					
2. Method blank done per prep batch and < RL or CRDL (CLP)?					
3. MS run at required frequency and within limits?					
4. MSD or DU run at required frequency and RPD within SOP limits?					
D. Other					
1. Are all nonconformances documented appropriately?					
2. Current IDL/MDL data on file?					
3. Calculations and transcriptions checked for error?					
4. All client/project specific requirements met?					
5. Date/Time of analysis verified as correct?					

General Comments:_____

Analyst & Date: _____ Second-Level Review & Date: _____

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APPENDIX C

MSA GUIDANCE

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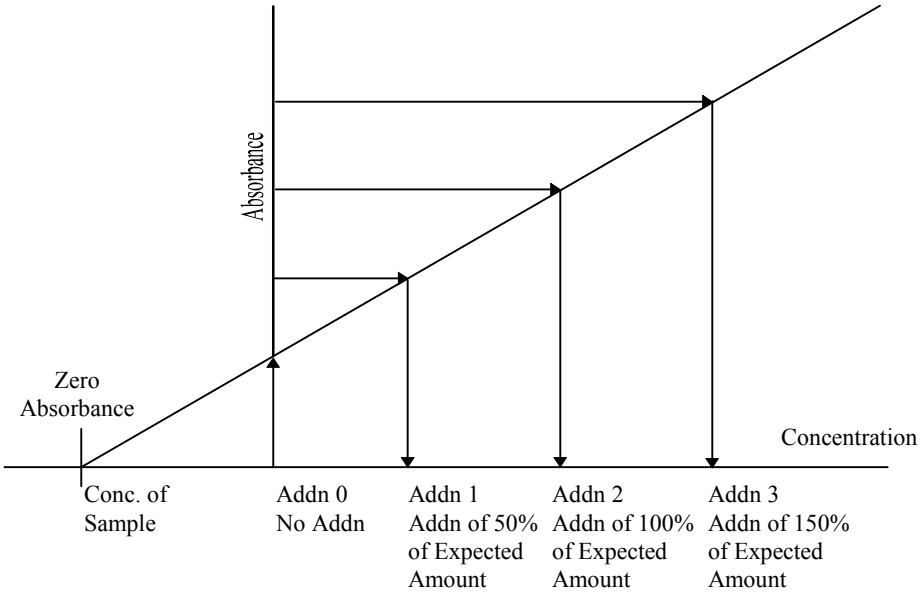
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



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- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

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APPENDIX D

PARTS MAINTENANCE GUIDE

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APPENDIX D. PARTS MAINTENANCE GUIDE

Maintenance Schedule

The software offers a simple to use online Scheduled Maintenance page. To view the page go to Instrument:Scheduled Maintenance (F1 Menu, I, S). A page displaying all items necessary to keep the instrument well maintained is shown (see figure 6.1A).

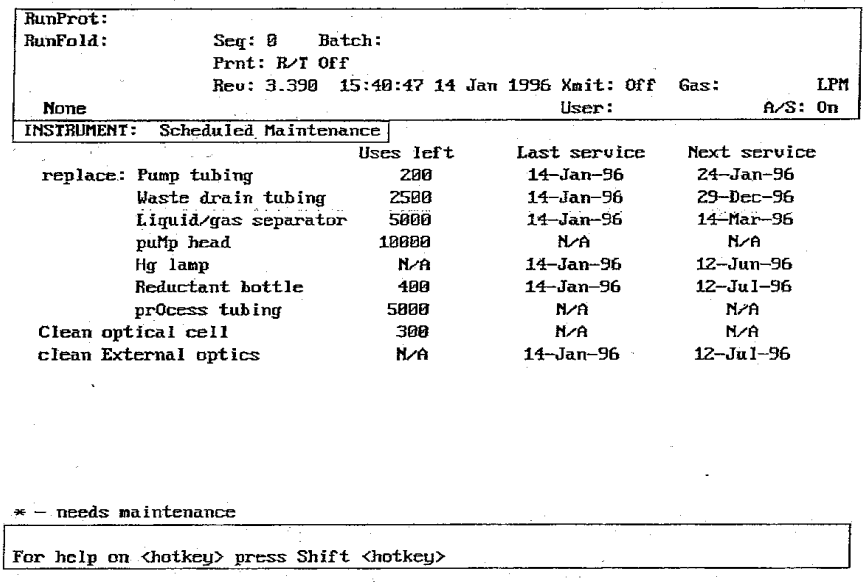

Figure 6.1a. Scheduled maintenance screen

Each scheduled maintenance item has a usage counter, timed usage, or both (N/A indicates that the usage counter or the timed usage is not applicable for that item). If either condition expires for a given item a maintenance message will alert the user at the top of the status box.

Maintenance Procedures

An asterisk(*) will appear next to the item requiring maintenance on the Scheduled Maintenance screen. To clear the message hit <Tab> or replace, clean, or replenish the item using the hot key for the item on the Scheduled Maintenance page. To perform the maintenance on a given item simply type the hot key (e.g. Type <P> for Pump tubing) and follow the directions. Once the directions are followed to completion, the usage counter and timed usage gets updated.

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APPENDIX E

CONTAMINATION CONTROL GUIDELINES

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APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 20% nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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APPENDIX F

PREVENTIVE MAINTENANCE

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APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200 II and Hydra AA)

Daily	Semi-annually	Annually
Clean lens windows with methanol.	Check Hg lamp intensity.	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check argon flow.		
Check tubing and replace, if needed.		
Check drain.		
Replace drying tube.		

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STL

Implementation Date: 5/7/07

STL PITTSBURGH STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY BY SW846 METHOD 7471A

(SUPERSEDES: PITT-MT-0007, REVISION 5)

Prepared by: Erin M Bozik

Reviewed by: Technology Specialist

Approved by: Quality Assurance Manager

Approved by: Environmental Health and Safety Coordinator 5-7-07

Approved by: Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A.

1.2. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.3. Method 7471A is applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, sludge-type materials wipes, and tissue matrices. All matrices require sample preparation prior to analysis.

1.4. The STL reporting limit for mercury in solid matrices is 0.033 mg/kg based a 0.6 g sample aliquot (wet weight). The STL reporting limit for wipes is 0.02 ug/wipe.

1.5. For DoD QSM Version 3 requirements, refer to SOP PITT-QA-DoD-0001.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride 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. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Total Metals: The concentration determined on an unfiltered sample following digestion.

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4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Potassium permanganate, which is used to breakdown organic mercury compounds, also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. 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 at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

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4.6. The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. 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.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 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/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.
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.

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Potassium Permanganate	Oxidizer	5 Mg/M3 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.
Stannous Chloride	Irritant	2 Mg/M3 TWA as Tin	Causes irritation to the respiratory tract. Can irritate skin and eyes. Symptoms include coughing and shortness of breath. Contact with skin and/or eyes may cause redness, itching and pain.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since mercury vapor is toxic, precaution must be taken to avoid its inhalation,

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ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.5.1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4, or

5.5.2. Iodine, 0.25%, in a 3% KI solution.

5.6. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.7. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.8. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.9. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

5.10. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor and/or the EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath (capable of maintaining temperature of 90- 95 °C) or autoclave capable of obtaining 15 lbs., 120 °C.

6.2. LEEMAN Labs PS200II Mercury Analyzer:

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6.2.1. LEEMAN Mercury Lamp P.N. 317-00048

6.2.2. Peristaltic Pump

6.2.3. Flow Meter

6.2.4. Printer

6.2.5. Dehydrator tube

6.3. Leeman HYDRA AA Automated Mercury Analysis System.

6.4. Disposable Sealable Sample Containers (Corning).

6.5. Argon gas supply (ultrahigh purity-grade).

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Top-loading balance, capable of reading up to two decimal places.

6.9. Thermometer (capable of accurate readings at 95 °C).

6.10. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (1000 ppm) mercury standards (in 10% HNO_3) are purchased as custom STL solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Intermediate mercury standard (10 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The intermediate standard must be made monthly and must be prepared in a matrix of 2% HNO_3. This acid (2 mL of

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concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. Working mercury standard (0.1 ppm): Take 1 mL of the intermediate mercury standard (7.3) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot. A second source working standard is prepared at 0.1 ppm for preparation of the ICV.

7.5. The calibration standards must be prepared fresh daily from the working standard (7.4) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample prep bottles and proceeding as specified in Section 11.1. The 0, .5, 1.0, 5.0 and 10 standards are recommended by Thermo Electron. The 0.2 standard level was selected to include a standard at the RL. See Table 1 (Appendix A) for the preparation of the ICV, CCV and RLV standards.

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.6. The initial calibration verification standard (ICV) must be made from a different stock solution than that of the calibration standards.

7.7. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.9. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9.1. Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated H_2SO_4 to 1 liter with reagent water.

7.10. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

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7.11. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.12. Stannous chloride solution: Add 200 g of stannous chloride to 2 L of 10% hydrochloric acid.

Note: Stannous sulfate may be used in place of stannous chloride. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

7.13. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.14. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

7.15. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of sample analysis.

8.2. Soil and wipe samples do not require preservation but must be stored at 4° C ± 2° C until the time of analysis. Tissue samples are stored frozen.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7471A, the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, and have been carried through the entire analytical procedure. MDLs must be

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redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS and MS/MSDs) are not included in the sample count for determining the size of a preparation batch.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level). **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the method blank.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such**

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action must be taken in consultation with the client and must be addressed in the project narrative.

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. The CCV results can be reported as the LCS results since all CCVs (as well as all other standards) are processed through the sample preparation step with the field samples. No more than 20 samples can be associated with one CCV used for the purpose of reporting LCS data.

- If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the instance where the LCS recovery is > 120% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the case narrative.

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

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- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 75 - 125 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the MS/MSD.**

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the ICB.** If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples and at the end of the analytical sequence. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. The CCB result must fall within +/- RL from zero. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the CCB.** Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis must be terminated, the

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problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

9.9. Reporting Limit Verification Standard (RLV) – Calibration accuracy at the laboratory reporting limit is verified after the analysis of the ICB. Until in-house control limits are established, a control limit of 50 - 150% recovery will be applied.

9.10. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences, which cause a baseline shift. Refer to Section 11.2.12 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the STL reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

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10.6. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient.

10.7. Refer to Section 9.0 and Table II for calibration verification procedures, acceptance criteria and corrective actions. The NELAC requirement for verification of initial calibration at varied concentrations is met daily since the ICVs, CCVs, and RLVs are all at different concentrations.

11. PROCEDURE

11.1. Standard and Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB, RLV) are processed through the digestion procedure as well as the field samples.

11.1.2. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (7.5) into a series of sample digestion bottles.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.1.3. Add reagent water to each standard bottle to make a total volume of 10 mL. Continue preparation as described under 11.1.5 or 11.1.6 below.

11.1.4. Transfer triplicate, 0.2 g portions of a well mixed sample into a clean sample digestion bottle. Refer to PITT-QA-0024 for subsampling procedures. For wipes, add the entire contents of the sample jar into a clean sample digestion container. Continue preparation as described under 11.1.5 or 11.1.6 below.

11.1.5. Water Bath protocol (optional procedure):

11.1.5.1. To each **standard** bottle: Add 5 mL of aqua regia.
To each **sample** bottle: Add 5 mL of reagent water and 5 mL of aqua regia.

11.1.5.2. To the LCS, add 2.5 mL, and to the MS, and MSD, add 1.0 mL of the 0.1 ppm working standard (7.4).

11.1.5.3. Heat for 2 minutes in a water bath at 90 - 95 ° C.

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11.1.5.4. Cool.

11.1.5.5. Add 50 mL of distilled water.

11.1.5.6. Add 15 mL of potassium permanganate solution.

11.1.5.7. Add 8 mL of potassium persulfate solution, mix thoroughly.

11.1.5.8. Heat for 30 minutes in the water bath at 90 - 95 °C.

11.1.5.9. Cool.

11.1.5.10. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

11.1.5.11. To each **standard** bottle: Add 50 mL of reagent water.
To each **sample** bottle: Add 55 mL of reagent water.

11.1.5.12. Continue as described under Section 11.2.

11.1.6. Autoclave protocol (default procedure):

11.1.6.1. Add 5 mL concentrated of H_2SO_4 and 2 mL of concentrated HNO_3.

11.1.6.2. To the LCS, add 2.5 mL, and to the MS, and MSD, add 1.0 mL of 0.1 ppm working standard (7.4)

11.1.6.3. Add 5 mL of saturated potassium permanganate solution.

11.1.6.4. Add 8 mL of potassium persulfate solution.

11.1.6.5. Cover digestion bottle with aluminum foil or screw cap loosely applied.

11.1.6.6. Heat samples at 121 °C and 15 lbs. pressure for 15 minutes.

11.1.6.7. Cool.

11.1.6.8. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce excess permanganate.

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Note: Alternate final volumes may be used as long as the standards and sample are treated the same way and reagents are adjusted proportionally.

11.1.6.9. Make up to volume of 100 mL with reagent water.

11.1.6.10. Continue as described under Section 11.2.

11.2. Sample Analysis:

11.2.1. Refer to the SOP PITT-MT-0028 and the instrument manuals for detailed setup and operation protocols for the LEEMAN PS200II and Hydra AA.

11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.

11.2.3. Automated determination: Follow instructions provided by instrument manufacturer.

11.2.4. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. ug of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.5. All measurements must fall within the defined calibration range to be valid. When sample concentrations exceed the upper limit of the calibration curve, the samples will be diluted and reanalyzed (if possible) to bring them within calibration curve. When reported sample concentrations either exceed the upper limit of the curve (i.e. cannot be rerun) or fall below the reporting limit, the data will be qualified as estimated.

11.2.6. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.7. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

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11.2.8. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.9. The following analytical sequence must be used with 7471A:

Instrument Calibration
ICV
ICB
RLV
Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Methods 7471A.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.2.10. The following run sequence is consistent with 7471A and CLP and may be used as an alternate to the sequence in 11.2.10. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration
ICV
ICB
RLV or CRA*
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.
CCV
CCB

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Refer to the appropriate CLP SOP (CORP-MT-0008) for quality control requirements for QC samples.

* Refer to the CLP SOP for information on the CRA.

11.2.11. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

Appendix C provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and parts maintenance. For instrument troubleshooting, use the autodiagnostics software. If a the problem cannot be determined using the software, place a call to service personnel

11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

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12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. RLV percent recoveries are calculated using the same equation as the ICV or CCV (replace ICV or CCV with RLV in the above equations).

12.4. Matrix spike recoveries are calculated according to the following equation:

$$\%\,R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.5. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

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$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.6. For automated determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = \quad (C \times V \times D)/(W \times S)$$

Where:
C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.7. For automated determinations, the final concentration determined in wipe samples is calculated as follows:

$$ug/wipe = \quad (C \times V \times D)$$

Where:
C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor

12.8. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

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12.9. Sample results should be reported with up to three significant figures in accordance with the STL significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Method performance is determined by the analysis of method blank, laboratory control sample, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 25 % and the matrix spike duplicates should compare within 20% RPD. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the MS.** The method blanks must meet the criteria in Section 9.3. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the method blank.** The laboratory control sample should recover within 20% of the true value until in house limits are established.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. 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. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. The following waste streams are produced when this method is carried out.

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15.1.1. Extracted sample containing less than 1 ppb Hg. This waste is collected in waste containers identified as "Acid Waste", Waste #33. It is neutralized to a pH between 6 and 9 and is disposed down a lab sink.

15.1.2. Unused Standards. This waste collected in containers identified as "Mercury Standards Waste", Waste #4.

15.1.3. Extracted sample containing greater than 1 ppb Hg. This waste collected in containers identified as "Mercury Standards Waste", Waste #4.

15.1.4. Mercury Analyzer Waste. Waste discharged from mercury analyzer is collected in containers identified as "Mercury Standards Waste", Waste #4.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

16.2. U.S.EPA Statement of Work for Inorganics Analysis, ILM04.0.

16.3. QA-003, STL QC Program.

16.4. QA-004, Rounding and Significant Figures.

16.5. PITT-QA-007, Method Detection Limits.

16.6. PITT-QA-DoD-0001, Implementation of the DoD QSM Version 3.

16.7. PITT-QA-0024, Subsampling.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from 7471A.

17.1.1.1. A potassium persulfate oxidation step has been included to facilitate the breakdown of organic mercurials which are not completely oxidized by potassium permanganate. Use of potassium persulfate in combination with the permanganate improves the recovery of mercury from organo-mercury

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compounds. The use of persulfate has been incorporated in several recent EPA mercury protocols.

17.1.1.2. The alternate run sequence presented in Section 11.2.11 is consistent with method requirements.

17.1.1.3. Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.4. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.2. Modifications from Revision 4: Safety Section 5.0, Pollution Prevention Section 14.0 and Waste Management Section 15.0 updated. References to DoD criteria were added. Wipe samples were added to the SOP.

17.2. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Non-conformance summary (if applicable).

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Figure 1. Solid Sample Preparation for Mercury - Autoclave Procedure (Default)

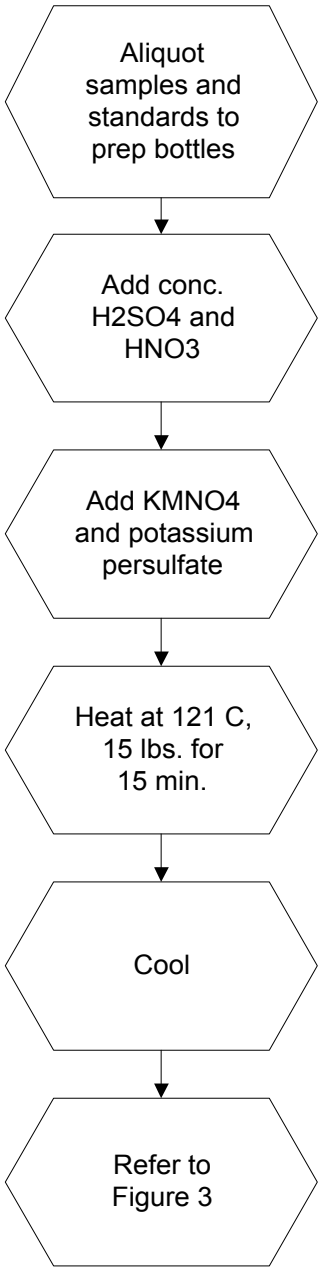

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Figure 2. Solid Sample Preparation for Mercury - Water Bath Procedure (Optional)

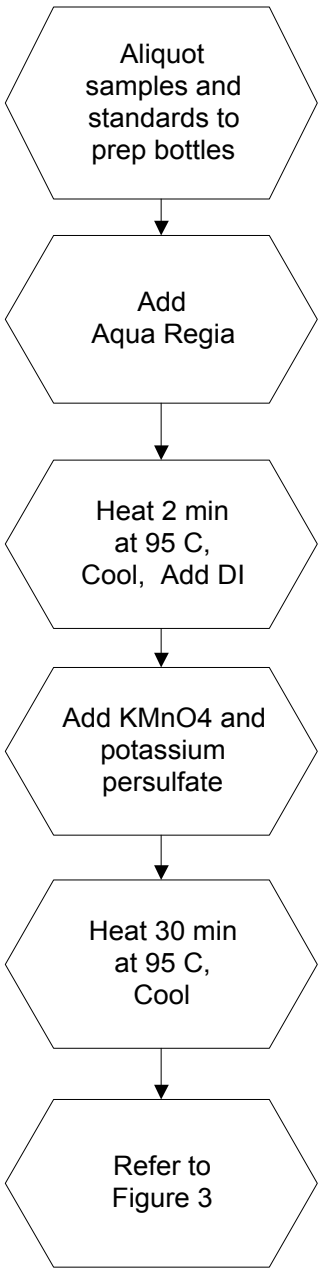

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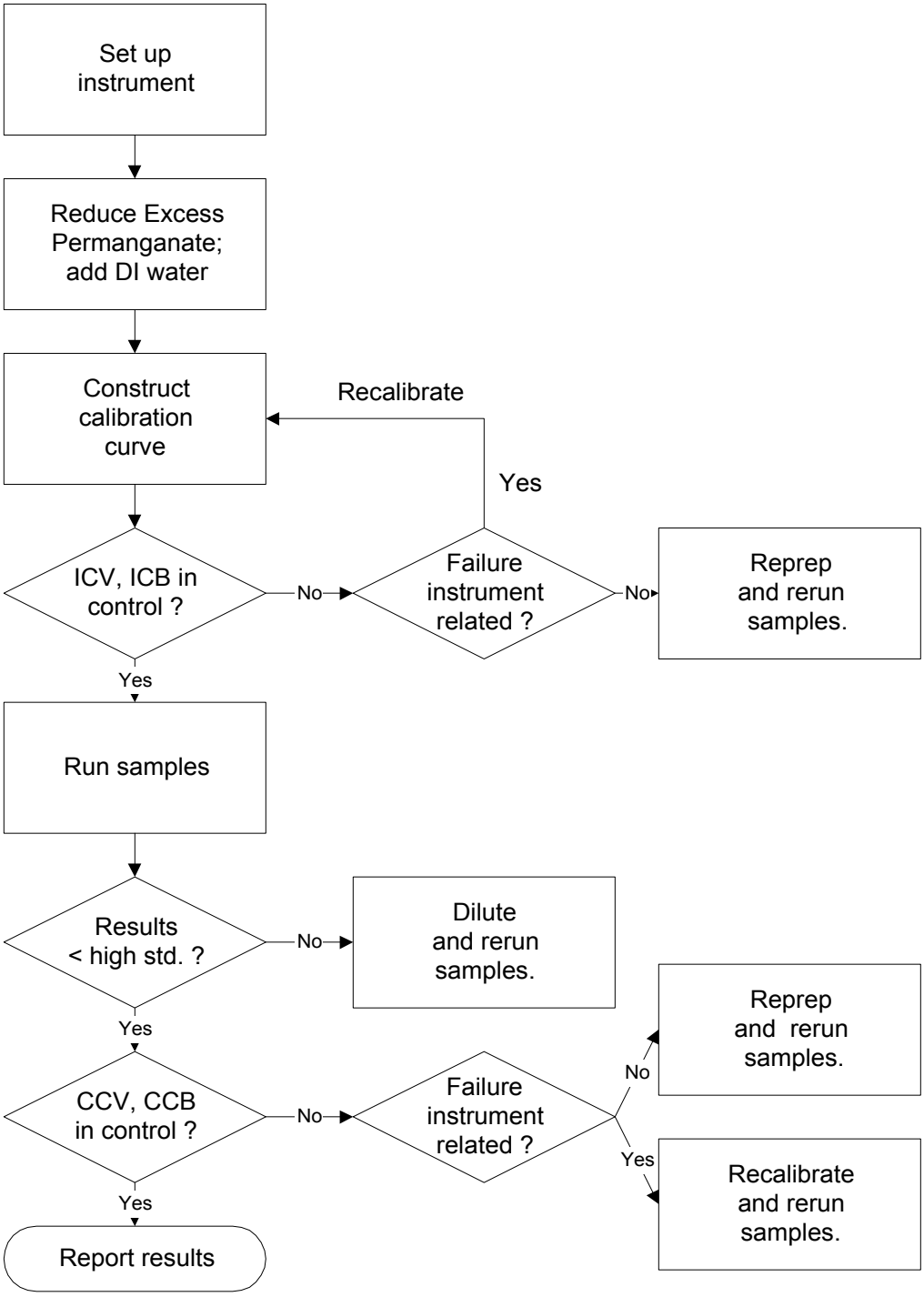
Figure 3. CVAA Mercury Analysis



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APPENDIX A

TABLES

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TABLE I . MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS

Method	**Reporting Limit**	
SW846 7471A	0.033 mg/kg or 0.02 ug/wipe	
Standard or QC sample	**mLs of 0.1 ppm Working Standard**	**Concentration (mg/L) *** **
Std 0	0	0
Std 1	0.2	0.0002
Std 2	0.5	0.0005
Std 3	1.0	0.001
Std 4	5.0	0.005
Std 5	10.0	0.010
ICV	2.5 **	0.0025
CCV	5.0	0.005
RLV	0.2	0.0002
LCS	2.5	0.0025
MS	1.0	0.001

*** SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.**

**** Prepared from a second source 0.1 ppm working standard.**

***** When brought to a 100 mL final volume.**

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TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.(1)	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.7).
RLV	Beginning of every analytical run, immediately following the ICB.	50 - 150 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.9).
CCV	Every 10 samples and at the end of the run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.8).
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.(1)	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep batch (see Section 9.8).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL.(1) Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.4)	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.

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TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits.(1) If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.6) For TCLP see Section 11.2.12
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD ≤ 20%.(1) (See MS)	See Corrective Action for Matrix Spike.

(1) For specific DoD requirements, refer to PITT-QA-DoD-0001.

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APPENDIX B
STL Hg DATA REVIEW CHECKLIST

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STL Pittsburgh Data Review Checklist – Mercury

Run Date: _____ Lots Analyzed: 4. _____ 8. _____ 12. _____

Analyst: _____ 1. _____ 5. _____ 9. _____ 13. _____

Instrument: _____ 2. _____ 6. _____ 10. _____ 14. _____

Methods: _____ 3. _____ 7. _____ 11. _____ 15. _____

Review Item	Yes (✔)	No (✔)	N/A (✔)	2nd Level Review (✔)	Comments
A. Calibration/Instrument Run QC					
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels?					
2. ICV/CCV analyzed at appropriate frequency and within control limits?					
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?					
4. CRA run? (CLP only)					
B. Sample Results					
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?					
2. All reported results bracketed by in control QC?					
3. Sample analyses done within holding time?					
C. Preparation/Matrix QC					
1. LCS done per prep batch and within QC limits?					
2. Method blank done per prep batch and < RL or CRDL (CLP)?					
3. MS run at required frequency and within limits?					
4. MSD or DU run at required frequency and RPD within SOP limits?					
D. Other					
1. Are all nonconformances documented appropriately?					
2. Current IDL/MDL data on file?					
3. Calculations and transcriptions checked for error?					
4. All client/project specific requirements met?					
5. Date/Time of analysis verified as correct?					

General Comments: _____

Analyst & Date: _____ Second-Level Review & Date: _____

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APPENDIX C

MSA GUIDANCE

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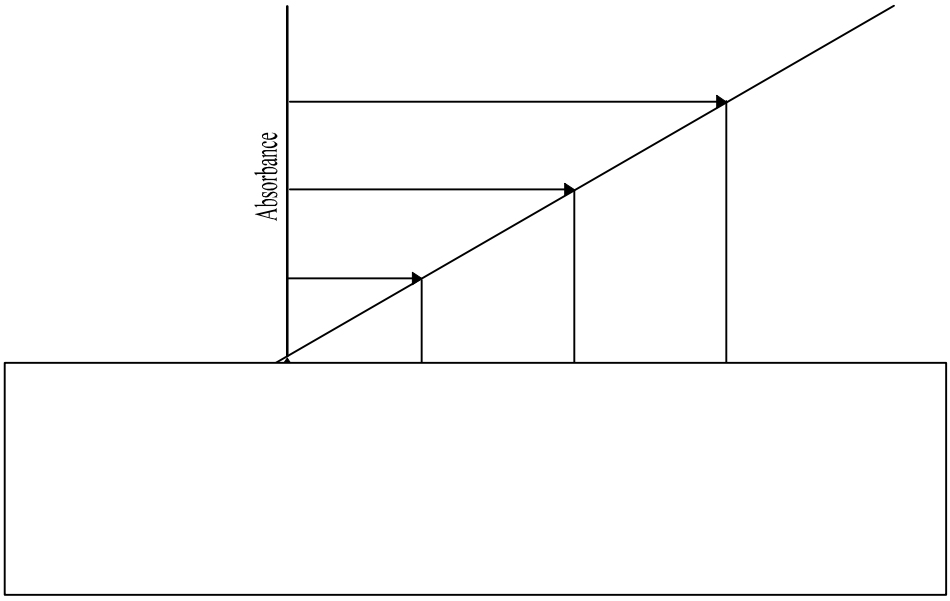
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



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- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

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APPENDIX D

PARTS MAINTENANCE GUIDE

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APPENDIX D. PARTS MAINTENANCE GUIDE

Maintenance Schedule

The software offers a simple to use online Scheduled Maintenance page. To view the page go to Instrument:Scheduled Maintenance (F1 Menu, I, S). A page displaying all items necessary to keep the instrument well maintained is shown (see figure 6.1A).

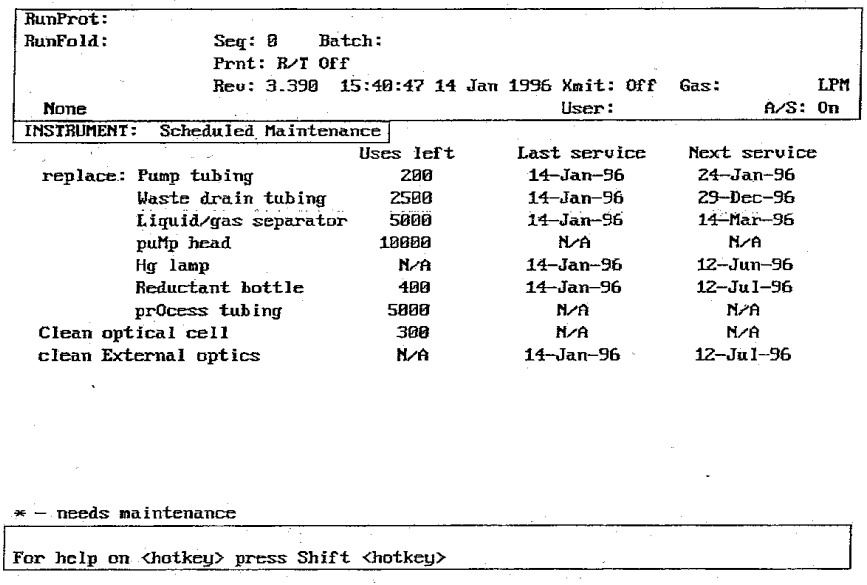

Figure 6.1a. Scheduled maintenance screen

Each scheduled maintenance item has a usage counter, timed usage, or both (N/A indicates that the usage counter or the timed usage is not applicable for that item). If either condition expires for a given item a maintenance message will alert the user at the top of the status box.

Maintenance Procedures

An asterisk(*) will appear next to the item requiring maintenance on the Scheduled Maintenance screen. To clear the message hit <Tab> or replace, clean, or replenish the item using the hot key for the item on the Scheduled Maintenance page. To perform the maintenance on a given item simply type the hot key (e.g. Type <P> for Pump tubing) and follow the directions. Once the directions are followed to completion, the usage counter and timed usage gets updated.

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APPENDIX E

CONTAMINATION CONTROL GUIDELINES

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APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 20% nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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APPENDIX F

PREVENTIVE MAINTENANCE

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APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200 II and Hydra AA)

Daily	Semi-annually	Annually
Clean lens windows with methanol.	Check Hg lamp intensity.	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check argon flow.		
Check tubing and replace, if needed.		
Check drain.		
Replace drying tube.		

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

TESTAMERICA PITTSBURGH STANDARD OPERATING PROCEDURE

TITLE: ANALYSIS OF METALS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY (ICPMS) FOR METHODS 200.8, 6020 & ILM05.2

(SUPERSEDES: REV 3.0, 1/30/07)

Prepared by: Erin M Bozik

Reviewed by: _____
Technical Reviewer

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____ 6/22/07
Lab Director

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS) by EPA Method 6020 and EPA Method 200.8.

1.2. This method is applicable to drinking, surface, and saline waters; soil, wipe, tissue and waste samples.

1.3. Reporting Limits

The standard reporting limits for metals analyzed by ICP-MS are listed in Table 1. Upon client request, results below the standard reporting limit but above the current method detection limit (MDL) may be reported and qualified as “estimated”.

1.4. Methods are based on the requirements of the US EPA Contract Laboratory Program (CLP) method ILM05.2D, and SW-846 methods 6020 and 6020. Instructions within this document that are general are given in BLACK, whilst those that apply only to 6020 are in BLUE and those that apply only to ILM05.2D are in RED.

1.5. Elements that may be determined using this procedure include: Al, Sb, As, Ba, Be, B, Cd, Cr, Co, Cu, Fe, Pb, Mn, Mo, Ni, Se, Si, Ag, Sr, Tl, Sn, Ti, V, Zn, Ca, Mg, K, P, Cs and Na.

Note: successful Ag analysis may require all solutions to be prepared as described, but with the addition of hydrochloric acid to 1% (v/v). This may degrade performance for As, Se and V.

1.6. For DoD QSM Version 3 requirements, refer to SOP PITT-QA-DoD-0001.

2. SUMMARY OF METHOD

2.1. The sample solution is introduced into a pneumatic nebulizer via a peristaltic pump. The nebulizer generates a fine aerosol by bringing the solution into contact with a high velocity flow of argon gas at its tip. The nebulized sample is sorted by droplet size in the spray chamber. Large droplets are rejected, whilst smaller particles are transported with the gas stream into the plasma.

2.2. The argon plasma operates with a continuously applied radio frequency (RF) field to give a high-energy discharge consisting of argon atoms, ions and electrons. The hottest part of the plasma can attain 6000-8000 K. In the plasma, aerosol droplets undergo evaporation, atomization and ionization. Ions are sampled through an aperture in a metal cone (sampler) at atmospheric pressure, into the expansion region at about 2 mbar and subsequently through an aperture in a second metal cone (skimmer) into the intermediate chamber.

2.3. An electrostatic ion lens system focuses the ion beam through a differential aperture into the analyser chamber, at about 10-7 mbar. The ions are filtered by mass-to-charge ratio in microsecond timescales by the quadrupole. The selected mass is detected by a discrete dynode electron multiplier. The multiplier has two simultaneous modes of operation: pulse count and analogue. The combination of these two modes allows seamless detection spanning 8 - 9 orders of magnitude. A detector “cross-calibration” is required for the analogue counts to be converted to equivalent pulse counts. The output from the detector is proportional to the concentration of the element in the aspirated solution, hence the concentration of unknown samples may be calculated when the instrument response is calibrated with standards of known concentration.

2.4. The linear range may vary from instrument to instrument and is dependant upon the sensitivity determined by the optimization parameters. This should be determined by the individual laboratory. In the test study at TestAmerica Pittsburgh, the linear ranges listed in Table 1 were obtained:

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2.4.1. Table 1. Test study linear ranges for the X5 ICP-MS

Analytes	Linear Range (mg/L)
Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl, V, Zn	0.20 – 20.0
Al, Ca, Mg, K Na, Fe	100 - 1500

2.5. Calibration standard concentrations are listed in Table 2.

2.5.1. Table 2. Calibration standard concentrations for analysis of water and waste

Analytes	Calibration Range (mg/L)
Al, Mn	1.0
Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Ni, Se, Ag, Tl, V, Zn	0.20
Ca, Mg, K Na, Fe	100
Fe	50
B, Mo, Sn, Sr, Ti	0.20
Si	10

3. DEFINITIONS

3.1. See the LQM for definitions of general terms

3.2. See appendix for Glossary of Abbreviations

4. INTERFERENCES

4.1. Isobaric interferences. Elemental isobaric interferences occur when different elements have isotopes at the same nominal mass, e.g. ^{114}Cd and ^{114}Sn. Problematic elemental isobaric interferences for these methods are listed in Table 3. The correction factors given in Table 3 are based on theoretical isotopic abundance ratios and may require adjustment.

Table 3 Isobaric Interferences and Correction Equations

m/z	Analyte	Interferent	Correction
58	Ni	Fe	58Ni=58M-0.0040*56Fe
64	Zn	Ni	64Zn=64M-0.0440*60Ni
82	Se	Kr	82Se=82M-1.0010*83Kr
114	Cd	Sn	114Cd=114M-0.0270*118Sn
115	In	Sn	115In=115M-0.0140*118Sn
123	Sb	Te	123Sb=123M-0.1240*125Te
138	Ba	Ce	138Ba=138M-0.0030*140Ce

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4.2. Abundance Sensitivity - Abundance sensitivity is the ability of the quadrupole to separate a low intensity peak from an adjacent high intensity peak. An example of the requirement of this is the detection of low concentrations of manganese (m/z 55) in the presence of high concentrations of iron (m/z 56). Quadrupole resolution and bias can be adjusted during set-up to resolve these signals.

4.3. Isobaric Polyatomic Ion Interferences - Polyatomic ions are produced by chemical reaction in the plasma and the interface region. If these polyatomic ions have the same nominal mass to charge (m/z) ratio as an analyte a polyatomic interference is observed. The principle polyatomic species for this method are listed in Table 4. Some of the correction factors given in Table 4 are based on theoretical isotopic abundance ratios and may require adjustment. Other factors were derived empirically. The stability of the empirical factors was determined during the test study at Thermo Electron. It was found that the factors require little or no adjustment and can be transferred between similarly configured X5 instruments.

Table 4. Isobaric Polyatomic Interferences and Correction Equations

m/z	Analyte	Interferent	Correction
51	V	ClO	51V = 51M-3.0460*53ClO 53ClO = M53-0.114*52Cr
52	Cr	ArC, ClOH	52Cr = 52M-0.0050*13C
56	Fe	CaO	56Fe = 56M-0.1500*43Ca
56	Co	CaO, CaOH	59Co = 59M-0.0046*43Ca
60	Ni	CaO	60Ni = 60M-0.0020*43Ca
75	As	ArCl	75As = 75M-3.000*77ArCl 77ArCl = 77M-0.8000*82Se 82Se = 82M-1.0010*83Kr
111	Cd	MoO	111Cd = 111M-0.9820*108MoO 108MoO = 108M-0.712*106Cd

4.4. Physical Interferences - Physical interferences include transport effects, ionization effects and deposition effects in the sample introduction system, plasma and interface, which result in signal suppression and signal drift. Transport effects arise from variations in solution properties, e.g. viscosity or surface tension, which affect nebulization efficiency and aerosol droplet size. The concentration of dissolved matter will affect the ionization efficiency of the analytes in the plasma and will cause a mass-dependant suppression effect and contribute to space-charge effects. Dissolved matter may also condense on the cones, altering the ion beam profile. This normally manifests itself as a time-dependant downward signal drift. To reduce the severity of these effects it is advised that the total dissolved solids concentration of solutions aspirated should be limited to <0.05%. Samples known to contain higher dissolved solids concentrations should be diluted. Signal suppression and drift can be corrected, to a degree, with the use of internal standardization techniques. Since these effects can be mass-dependant and may be related to the ionization potential of the element, a multiple-element internal standard approach should be used.

4.5. Memory Effects - Memory effects occur when the signal for an analyte from a sample contributes to the signal of a subsequent sample. This effect can be severe for certain elements due to their physico-chemical properties, e.g. mercury. This effect is minimised by aspirating a wash solution between samples. A monitored wash can be used in order to ensure that analyte signals recover to the background level.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2. The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.3. 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.
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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.4. Eye protection that protects against splash, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded;

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other gloves will be cleaned immediately.

5.5. The waste pumped from the spray chamber is corrosive and must be handled with care, especially if large volume containers are used, as these may be heavy and awkward to carry. Empty the waste vessel daily to reduce the quantity that must be disposed each time and to keep weight to a minimum. Protective clothing, including hand and eye protection must be worn when handling this waste.

5.6. The wash solution is corrosive and must be handled with care. This solution must be prepared and stored in a vessel made of a robust acid-resistant material with a tight fitting lid that it is resistant to breakage if dropped. Large volumes of this solution will be heavy and may be awkward to carry. Ensure adequate provision for transporting the vessel, i.e. suitable handles on the vessel, minimum distance between the preparation area and the instrument. Use a cart to transport the vessel where necessary or ask for assistance in carrying.

5.7. Many of the concentrated metal standard solutions are toxic and must be handled with care. Skin and eye protection should be worn when handling and inhalation of vapours must be prevented.

5.8. Fumes generated by the plasma can be hazardous and must be removed from the laboratory with an extraction system as detailed in the X Series site planning guide. If the extraction system is faulty do not attempt to use the instrument. The extraction system should be inspected on a regular basis.

5.9. The plasma emits strong UV light and is harmful to vision.

5.9.1. *WARNING:* AVOID looking directly at the plasma.

5.10. The plasma is a source of radio frequency (RF) radiation and intense, ultra-violet radiation that can damage the eyes. This radiation is normally contained by the system, but operators must be aware of the dangers. The instrument must be properly maintained by qualified service personnel. Never attempt to defeat hardware interlocks – they are there for your safety.

5.11. <u>WARNING: People with pacemakers should not go near the instrument while in operation</u>. DIAZOMETHANE is an extremely toxic gas with an explosion potential. Since the explosion potential is catalyzed by imperfections in glass, generation of diazomethane must be carried out in glassware free from etches, cracks, chips, and which does not have ground glass joints. Solutions of diazomethane will be kept at temperatures below 90°C. Diazomethane must be generated and handled in a fume hood.
Note: Diazomethane has not been classified as a carcinogen under the current OSHA definition.

5.12. Should the plasma need to be extinguished in an emergency, open the torch box door. This will immediately cut-off the power to the plasma RF generator, extinguishing the plasma. After extinguishing the plasma, the torch, torch box, cones and cone housing may remain very hot for some time. Operators must be aware of this fact and allow cooling time prior to handling these components.

5.13. There are high voltage components inside the instrument. Routine maintenance does not require access to any of the electronic components. If an electronic fault is suspected, a qualified service engineer must be called. Do not attempt to tamper with electronic components yourself.

5.14. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and

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waste containers will be kept closed unless transfers are being made.

5.15. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.16. All work must be stopped in the event of a known or potential compromise to the health and safety of an associate. The situation must be reported immediately to a laboratory supervisor and/or the EHSC.

6. EQUIPMENT AND SUPPLIES

6.1. (2) X Series ICP-MSs fitted with Xi interface and Y-connector for on-line internal standard addition (supplied with this package).

6.2. (2) Cetac ASX-510 autosamplers.

6.3. Ultrapure water system capable of delivering de-ionized, polished water of at least 18 MΩ cm

6.4. Yellow/orange tab peristaltic pump tubes (~0.5 mm ID)

6.5. White/white tab peristaltic pump tubes (~1 mm ID)

6.6. A range of adjustable pipettes, such as Rainin pipettes. Adjustable pipettes with a capacity of 0.1 mL, 1 mL, and 10 mL are recommended. These must be calibrated regularly to ensure accurate volumes are delivered.

7. REAGENTS AND STANDARDS

7.1. General Reagents

7.1.1. **Laboratory Water** - All laboratory water used in these procedures must be of very high quality, purified with a reverse osmosis system and polished with an ion exchange system to give a final product of resistivity >18 MΩ cm.

7.1.2. **Hydrochloric Acid** (sp. gr. 1.18) - Hydrochloric acid must be at least Romil “SPA”, J.T. Baker “Instra Analyzed”, BDH/Merk “Analar”, Fisher “Optima” - grade or equivalent. Hazards – corrosive, causes severe burns.

7.1.3. **Nitric Acid** (sp. gr. 1.42) - Nitric acid must be at least Romil “SPA”, J.T. Baker “Instra Analyzed”, BDH/Merk “Analar”, Fisher “Optima” - grade or equivalent. Hazards – oxidising and corrosive, causes severe burns.

7.1.4. **2 % (m/v) Nitric Acid** - This reagent is used for the calibration blank, ICB, CCB, sample dilution and solution preparation. Add 5 mL of Conc of HNO3 to DI water and dilute to 250 mL

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are to be collected in plastic or glass containers.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2.

8.3. All soil and wipe samples must be refrigerated to $4^{o}C \pm 2^{o}C$.

8.4. Tissue samples are stored frozen until preparation.

8.5. The analytical holding time for metals by ICP-MS is 6 months.

8.6. Aqueous samples for total metals must be digested before analysis using an appropriate digestion procedure. Method 200.8 has its own digestion specifications that are followed by the laboratory. Method 3005A is used for total recoverable metals and dissolved and

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method 3010A is used for total metals by 6020. These are covered in the SOP PITT-IP-003. Upon consultation with the client dissolved samples can forego digestion to help prevent contamination when very low detection limits are required.

8.7. Soil, wipe, tissue and waste samples should be digested before analysis using an appropriate digestion procedure. Method 3050B of SW846 is the appropriate digestion procedure. The SOP for 3050B is PITT-IP-0002.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration IDC and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For new analytes an MDL study should be performed and calibration curve generated before analyzing any samples.

9.2. Control Limits

9.2.1. Control limits are utilized for matrix spikes and laboratory control samples (LCS). These limits must be reviewed at least annually against current data.

QC Type	200.8	6020	ILM05.2
LCS	85 – 115	80 – 120	80 – 120
MS	70 – 130	75 – 125	75 – 125
RPD	± 20	± 20	± 20

9.2.2. All LCS and MS recoveries must be entered into QuantIMS or other database so that accurate historical control charts can be generated. For tests without a separate extraction, matrix spikes will be reported for all dilutions.

9.2.3. Refer to the QC program document (QA-003) for further details regarding control limits.

9.3. Quality Control Batch

The batch is a set of up to 20 field samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike/matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify particular samples for MS/MSD, the batch may contain multiple MS/MSDs. See policy QA-003 for further definition of the batch.

9.4. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS may be processed, if precision data is required by the client. The LCS pair is then evaluated according to the MS/MSD RPD criteria.

9.5. Method Blank

One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried

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through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 10% of the measured concentration of that analyte in the associated samples, whichever is higher. Certain programs, such as USACE, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the reporting limit. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the method blank.**

- If the analyte is a common laboratory contaminant (copper, iron, zinc), the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than five times the RL. Such action must be documented in the NCM program.
- Re-preparation and reanalysis of any samples with reportable concentrations of analytes less that 10 times the value found in the method blank is required unless other actions are agreed with the client.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported. This must be documented in the NCM program.
- If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all positive results in associated samples are flagged with a "J", and appropriate comments may be made in a narrative to provide further documentation.

9.5.1. Refer to the QC Program document (QA-003) for further details of the corrective actions.

9.5.2. For samples which have not been digested or matrix matched, a CCB result is reported as the method blank. The CCB analyzed immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.5.3. Methodologies for MDL assessment are detailed in SW-846 Chapter 1, method 6020 and in 40 CFR Part 136 Appendix B.

9.6. Laboratory Control Sample (LCS)

9.6.1. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless otherwise requested by the client.

9.6.2. If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur:

- Check calculations,
- Check instrument performance,
- Reanalyze the LCS, and if still outside of control limits,
- Evaluate the data, and/or
- Re-prepare and reanalyze all samples in the QC batch.

9.6.3. Data may be reported with an anomaly in the following cases:

- The LCS recoveries are high and the analyte of concern is not detected in field samples,
- All target requested analytes are within control, but other LCS compounds are out of control,
- If no sample preparation is performed (eg, dissolved metals), the LCS may be reprepared and reanalyzed within the same sequence.

9.6.4. The analyst should evaluate the anomalous analyte recovery for possible trends.

9.6.5. If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.

9.6.6. If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6.7. For samples which have not been digested or matrix matched, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same analytes as the LCS (See Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the historically generated limits. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the MS.**

Note: Some programs require a Matrix Spike and Matrix Replicate in lieu of an MS/MSD. When a matrix spike/matrix replicate is performed the matrix spike is evaluated for accuracy (% recovery) and the matrix replicate is evaluated for precision (RPD).

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.
- If the recovery for any component is outside QC limits for both the matrix spike/spike duplicate and the LCS, the process is out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If a MS/MSD or MS/Dup is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.
- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7.1. If the amount of an analyte found in the unspiked sample is greater than 4 times the amount of spiked analyte added, then routine control limits do not apply and recoveries are not evaluated. Other analytes in the MS and MSD must still be reported. File an NCM stating that the 4X rule was applied, and report the recovery in the LIMS as “ND MSB”. This NCM must be included in the final

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report.

9.7.2. For samples which have not been digested or matrix matched, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample.

9.8. Linear Range Verification (LR) - The linear range is determined semi-annually (2x/year) for each element on the standard list. See Section 13 for details of the linear range verification. The Linear Range study must be performed quarterly if doing ILM05.2.

9.9. The internal standard intensities in samples must be within 60 to 125% of the IS intensities for the Calibration Blank for method 200.8 and from 30% to 120% for method 6020. If this criterion is not met, the sample will be diluted and reanalyzed until the IS recoveries are within the limits. If the upper control limit is exceeded, the analyst should review the data for the presence possible contribution from the native sample. Narrate any findings.

9.9.1. For method 6020 the internal standard intensity in the ICV, ICB, CCV and CCB should be within 20% of the IS intensity in the calibration blank of the initial calibration. If not, the analyst should check for any instrument anomalies and continue if none are noted. For method 200.8 the IS acceptance range does not vary from the 60 to 125% noted above.

9.10. Interference Check Solutions (ICSAs) - The results of ICSA must be within ±3CRQL of the analytes “true” value or ±20% of the analytes “true” value, whichever is the greater. The “true” value will be taken as zero, unless otherwise indicated in the solution manufacturer’s literature. The software automatically checks for compliance with the above, based on a “true” value of zero. If a result falls outside this range, the analysis must be terminated and the samples associated must be reanalyzed. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the ICSA.**

9.11. Interference Check Solution Spike Recoveries (ICSABs) - Results of ICSAB must be within ±20% of the analytes “true” value. The software automatically checks for compliance with the above, based on the values indicated in (6.5.2 or 6.5.4). If a result falls outside this range, the analysis must be terminated and the samples associated must be reanalyzed.

9.12. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV must fall within ± 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within ± the reporting limit (RL) from zero. (Certain programs, may require a more stringent evaluation of ICB, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the reporting limit.) **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the ICB.** If either the ICV or ICB fail to meet criteria, the analytical sequence should be terminated, the problem corrected, the instrument recalibrated and the calibration re-verified.

9.13. CRQL Check Standard (CRI)

FOR ILM05.2, THE RESULTS OF THE CRI MUST BE WITHIN THE RANGE 70-130% RECOVERY FOR ALL ANALYTES, EXCEPT CO, MN AND ZN, WHICH MUST BE IN THE RANGE 50-150% RECOVERY. THIS IS CHECKED BY THE SOFTWARE, BASED ON THE VALUES GIVEN IN (6.6.3). IF ANY ANALYTE IS OUTSIDE THE RANGE INDICATED, THE SAMPLE MAY BE RE-RUN ONCE. IF THE RESULTS FALL WITHIN THE REQUIRED VALUES UPON RE-RUN, NO FURTHER CORRECTIVE ACTION NEED BE TAKEN. IF STILL OUTSIDE THE ACCEPTABLE RANGE, THE ANALYSIS SHALL BE TERMINATED, THE PROBLEM CORRECTED AND THE SAMPLES REANALYZED. **FOR NON-CLP METHODS** THE METHOD DOES NOT SPECIFY CRITERIA, HOWEVER THE LAB USES THE RANGE 50 – 150%. **REFER TO PITT-QA-DOD-0001 FOR SPECIFIC DOD REQUIREMENTS FOR THE CRI STANDARD.**

9.14. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. Results for the CCV must be within the range 90-110% recovery. This is checked by the software, based on the values in (6.6.2). If outside this range, the analysis must be terminated, the problem corrected and the samples since the last valid CCV must be re-analyzed. The CCB result must fall within ± RL from zero. (Certain programs, may require a more stringent evaluation of the CCB, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the reporting limit. The analyst should refer to the project notes provided by the PM to identify when this is an issue and if so what the corrective actions to take for exceedances.) **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the CCB.** Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, the CCV or CCB may be reanalyzed once and accepted if there is a reason for the initial out-of-control event such as carryover from a high concentration sample. Otherwise, if the CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to Section 11.9 for an illustration of the appropriate rerun sequence).

9.15. Post-Digestion Spike Samples (PDS) **For DoD samples, a post digestion spike will be run on a sample if the if the MS/MSD for the sample falls outside of % recovery criteria.** A post digestion spike is a matrix spike on a sample, which is added after the sample preparation is completed. For 6020 the default matrix spike protocol is a “post digestion spike”. However, TestAmerica Pittsburgh will perform a conventional matrix spike and spike duplicated as the default matrix QC. We will perform the “PDS” only where the conventional matrix spike fails. We believe that this approach will provide more complete matrix information than the default requirements. The spike recovery from the post digestion spiked sample should be within the range 75-125% where the spike value is greater than 25% of the indigenous analyte concentration. The software calculates this based on the following equation:

%Repeatability = 100 * (Spk-Orig)/Tru

where, Spk is the spiked sample result and Orig is the original sample result and Tru is the True spiked concentration value.If a result is outside the required range, the data should be assessed carefully and samples may require reanalysis.

9.16. Serial Dilution Samples (SER) - Some regulatory programs such as require a dilution test be performed for each matrix within an analytical batch determination. The results of the serial dilution sample(s) (SER) after dilution correction should be within the range 90-110% of the original sample, if the result for the original sample is greater than 50*IDL for CLP or greater than 50*MDL for 200.8 or 6020.

9.17. The software calculates this based on the following equation:

9.18. %Repeatability = 100 * Ser/Orig

9.19. where, Ser is the dilution corrected serial diluted sample result and Orig is the original sample result. If a result is outside the required range, the data should be assessed carefully and samples may require reanalysis.

9.20. Duplicate Samples (DUP); %RPD = ±20%: Results of the duplicate sample(s) (DUP) must be within ±20% of the results of the original sample, where the result is greater than or equal to 5*CRQL for CLP or greater than 5*RL for 200.8 or 6020. The software calculates this based on the following equation:

%RPD = (S-D) / [(S+D)/2] * 100%

where, D is the duplicate sample result and S is the original sample result.

If a result is outside the required range, the data should be assessed carefully and samples affected may need to be reanalyzed where the project requires it.

9.21. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the QA Manager.

9.22. Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.23. QC Program

Further details of QC and corrective action guidelines are presented in the QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

10.1. Instrument start-up

10.1.1. Follow the instrument start-up procedure outlined in the Thermo X-Series ICP-MS Operator's Manual.

10.2. Instrument Tuning

10.2.1. Aspirate a 20 ppb tuning solution containing all of the tuning elements. The 6020 tuning elements are Li, Co, In, and Tl. The instrument manufacturer monitors Mg, Ce, Be & Pb for instrument performance.

10.2.2. Mass calibration and resolution checks must be documented and included as part of the raw data package.

10.2.3. Resolution must be < 0.90 amu at 10% peak height for the 6 tuning (Be, Ce, Co, In, Mg, & Pb) for 6020. Resolution must be ≤ 0.75 amu at 5% of the peak height for ILM05.2. And the resolution must be ≤ 0.9 amu at 5% of the peak height for Method 200.8.

10.2.4. Mass calibration must be within ± 0.1 amu from the actual value for the 6 tuning elements (Be, Ce, Co, In, Mg, & Pb) or the mass calibration must be adjusted.

10.2.5. A "daily" performance check must be performed. This uses the same tuning solution as above. The 6 tuning elements must have RSDs below 5%. The oxides

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must be below 3.5%. If any of these conditions are not met repairs or optimization procedures must be performed until these specifications are met.

10.3. Initial Calibration

10.3.1. Calibration consists of a blank and the following calibration standards (STD1, STD 2X, and STD 3X see Table 2 for concentrations) in accordance with the manufacturer's procedure. Use the average of three integrations for both calibration and sample analyses.

10.3.2. Following the STD, STD2X & STD3X, an ICV/ICB pair is analyzed. The ICV must be within ± 10% of the true value to be acceptable.

10.3.3. For 6020 and ILM05.2, following the ICV/ICB pair, the CRI/RLV is run then the ICSA is analyzed.

10.3.4. For 6020 and ILM05.2, following the ICSA, analyze the ICSAB. The ICSAB must be within ± 20% of the true value.

10.3.5. Internal standards are added to all standards and samples by the instrument automatically prior to analysis.

10.4. Continuing Calibration:

10.4.1. Following every 10 samples (including lab QC), analyze a CCV/CCB pair. These must be within ± 10% of the true value for analysis to continue. For methods 6020 and ILM05.2, a CCV/CCB pair should also be analyzed immediately after the ICSAB.

10.4.2. All samples must be bracketed by an acceptable CCV/CCB pair. Where a CCV/CCB fails the samples preceding it back to the last acceptable CCV/CCB must be reanalyzed.

11. PROCEDURE

11.1. Instrument Set-up

11.1.1. Configure the X Series with the standard sample introduction equipment, i.e. a glass concentric nebulizer, glass impact bead spray chamber and a one-piece torch with 1.5mm ID injector tube. A Peltier spray chamber cooling unit is optional. Ensure that the Xi interface cones are fitted. These are standard with the X5 instrument and an option for the X7. They can be identified as follows:

Xi Sampler - 1.1 mm orifice, no nipple, no holes around the flat circumference

Xi Skimmer - Small pointed skimmer mounted in a copper adapter with two screws

Yellow/orange tab peristaltic pump tubes (5.2.6) should be used for sample and internal standard uptake. Connect the liquid output end of the peristaltic pump tubes to the 1.0 mm (OD) barbed fitting screwed into the Y connector. Note that the barbed fitting may require tightening with a pair of grips to ensure a good fluid-tight seal. The mixed output flow should be connected to the nebulizer. See diagram in Appendix 6 for plumbing schematic. A white/white tab peristaltic pump tube (5.2.7) should be connected to the spray chamber drain outlet at one end and to a tube running into a waste vessel at the other and wound on the pump to draw the waste liquid away from the spray chamber.

11.1.2. Perform the daily maintenance as outlined in Appendix 3.

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11.1.3. Switch the instrument into the *Operate* state by clicking the *ON* button at the top of the screen. During the automated ignition sequence, the following processes occur:

i. Torch purge with argon gas

ii. RF power match

iii. Plasma ignition

iv. Slide valve open

v. Electronics on

This process takes about two minutes. Upon successful ignition, the software will display *Operate* in the *Instrument State* bar. If the event of unsuccessful ignition, the software will display an error message and/or place a message in the *Technician Event Log*. Upon unsuccessful ignition, inspect the sample introduction equipment and torch, ensuring a good gas-seal at each connection and ensuring the torch is not misaligned or damaged. If all appears satisfactory, the ignition may be attempted again. If the ignition process consistently fails, contact your local Thermo service agent for advice.

11.1.4. Once the instrument is in the *Operate* state, it should be left for 30 minutes to reach thermal equilibrium prior to starting analytical measurements. The optimization (tuning), performance testing and instrument set-up calibrations may be performed after 15 minutes. Ensure that the peristaltic pump is operated at a default analytical speed of 15%. This is done by clicking on *Instrument*, *Configurations*, *Configuration Editor*, *View Selected Accessories* (network icon), *Peristaltic Pump*, *Connect* (chain icon). Set pump speed to 15% using the slider bar and adjust the *Settle Time* to 10 seconds and click on *Apply*. Click *OK* to close the dialogue box.

11.1.5. During the initial 15 minutes, the system can be “conditioned” by aspirating the system thoroughly with 2% nitric acid + 1% HCL solution (6.1.4) prior to continuing.

11.1.6. Instrument tuning (optimization) is performed using a 20 µg/L Tune Solution (6.4.1), aspirated through the sample uptake tube. Optimization may not be necessary from day to day if the sample introduction system and cones have not been adjusted in any way and if the instrument fulfils the performance requirements given below. If the instrument gives performance exceeding the requirements shown below, proceed to 8.1.7. Otherwise, tune the instrument manually or using *Autotune* while aspirating 20 µg/L Tune Solution (6.4.1) through both the sample and internal standard uptake tubes. *Autotune*, using an appropriately defined sequence is advised (see Appendix 4).

The final conditions must give the following:

^{9}Be	>2000cps
^{115}In	>50000cps
^{208}Pb	>25000cps
$^{156}CeO/^{140}Ce$	<0.02

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If the above criteria are met, proceed to 8.1.7. If the above criteria are not met, do not proceed. Check that the tune solution was prepared as per instructions in (6.4.1) and remake if necessary. If the sensitivity is below the minimum requirement, a new detector plateau may be required (see Appendix 6), the cones may require cleaning (see Appendix 8), or the nebulizer or sample uptake lines may have become blocked or may not be properly clamped on the peristaltic pump. If the CeO/Ce ratio is >0.025, the nebulizer gas flow can be reduced and/or the sampling depth increased, obtaining a corresponding reduction in oxide formation. Recheck the above parameters after taking any remedial action.

11.1.7. Save the satisfactory instrument settings by clicking on the disk icon on the Tune page. Note that this is not necessary if Autotune has been used, as the instrument settings are saved automatically (unless manual adjustments have been made after autotuning).

11.1.8. Set-up the resolution as described in Appendix 5.

11.1.9. Perform a cross-calibration (and mass-calibration and detector voltage setup if required) as explained in Appendix 6. Note that retuning may be necessary after performing this routine.

11.1.10. Aspirate Tune solution (6.4.1) and run a *Performance Report* (see Appendix 4) to confirm the mass-calibration, resolution, minimum sensitivity and maximum cerium oxide requirement given in (8.1.6) and to verify instrument stability. The performance report acquires five consecutive one-minute runs and calculates the percentage relative standard deviation (RSD) of the five measurements for each isotope. The RSD of the elemental analytes in the performance report must be <5%. If the performance report passes, proceed to (8.1.11). If the performance report fails, check:

a. Liquid uptake tubes for kinks or other damage

b. Condition and position of the peristaltic pump tubing

c. Tightness of the peristaltic pump clamp screws (these should be just tight enough to draw liquid through the tube smoothly)

d. Joints of all sample introduction components, ensuring a good seal

e. Nebulizer for blockage

f. Salt deposition on cones

Remedy the above as necessary and repeat the test. Note that retuning may be required if any sample introduction components are adjusted or replaced.

Note: Resolution set-up may require adjustment if the resolution check fails (see Appendix 5). Note that the quadrupole and hexapole bias strongly influence abundance sensitivity (Pole Bias should be kept >+4V and Hexapole Bias <-3V).

If the measured mass position for each mass in the performance report is not within ±0.1 amu of the nominal mass position, a new mass-calibration must be performed (see Appendix 6).

11.2. Sample Analysis

11.2.1. Open the method template by clicking on *Templates* and then <TESTAMERICA PITTSBURGH ICPMS ANALYSIS>. The method template will be opened. This contains all the saved analytical parameters and only the sample list need be amended.

11.2.2. Go to *Sample List*. This grid contains all the information about calibration, QC and samples to be run. The calibration and QC concentration information is already stored. Enter all unknown samples into the list in the appropriate order below the existing calibration and QC samples by overwriting the sample label fields. Delete any QC samples that do not apply to the required method. (If sample list changes are to be made permanent to the method, save the method as a *Template*, by going to *File*, *Save as Template*. Enter a new name to create an amended method, or use the same name to overwrite the current one.)

11.2.3. Once all the sample information is added, check the required autosampler positions have been correctly entered. Amend as necessary. To sequentially renumber positions, add the correct position required for the initiation of the sequence and right mouse click on the first correctly numbered cell. A pop-up menu will appear. Select *Renumber autosampler positions* from this. Ensure that all samples have one survey run and 3 main runs and a probe depth of 155mm.

11.2.4. Save the experiment run by clicking on the *File* menu, then *Save as*. Enter the required file name, e.g. *enviro090902* and click *Save*.

11.2.5. To print the sample list, go to *Reports* and check the *Sample List* box. Click the refresh icon. The sample list will be displayed in a printable format. Press the print icon. Note that this can only be done with PlasmaLab version 2.3 and above.

11.3. Loading the Autosampler

11.3.1. Pour the required samples into pre-cleaned 15ml polypropylene test tubes (5.1.4). To avoid contamination, a small amount of the solution to be analyzed can be poured into the tube and then discarded. This will rinse out any residual contamination.

11.3.2. Pour blanks, standards and QCs (positioned in rack 0) into pre-cleaned 50ml polypropylene tubes (5.1.5). To avoid contamination, a small amount of the solution to be analyzed can be poured into the tube and then discarded. This will rinse out any residual contamination. Note that **2% nitric acid** (6.1.4) is used as the calibration blank, ICB, and CCB.

11.3.3. For the **serial dilution** ("L") sample(s), dispense 2.00±0.02 mL of the original sample into a pre-cleaned 15 mL polypropylene test-tube (5.1.4) and add 8.00±0.08 mL of 2% nitric acid (6.1.4). Mix well. This is a 5-fold dilution.

11.3.4. Place the tubes for each sample into the appropriate position in the rack according to the sample list. Note that the autosampler works on a two-dimensional grid position system by rack number (0-4). See Appendix 9 for autosampler position map.

11.4. Initiating Analysis

11.4.1. Place the sample probe into the autosampler arm and the internal standard probe into the internal standard solution (6.4.6).

11.4.2. Go to *Instrument*, *Tune* and click on the accessories dialog icon. Click on *Autosampler* and then on the chain icon to connect. The autosampler should initialize. Ensure that the probe is at the correct height by positioning it so that its tip just protrudes through the hole in the bottom of the arm. Click on the *Go to Wash* icon (faucet) to send the probe to the wash station. Ensure that the wash solution is being correctly delivered to the wash station via the peristaltic pump at the rear of the autosampler. Allow at least 2 minutes for the liquid to be delivered

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to the sample introduction system.

11.4.3. Click on the experiment to be run. Click the *Queue* icon and then *Append* and *OK*. The analysis has now been initiated.

11.4.4. To monitor the progress of the analysis, right-mouse click on the *MS* icon at the bottom-right of the screen and select *Open Service Window* from the pop-up menu. The Service Window hovers over the current application window until moved or closed and displays the current instrument activity. This window is also used **to stop an analysis** if required. This is done by clicking on the ***^{x}Q* icon**.

11.4.5. To view results as they are generated, click on the experiment icon and go to the *Results* tab. Click on the *Refresh* button or the refresh icon (green circular arrows on a page) to calculate the results from the data obtained.

11.4.6. To view calibration plots, click on the *Calibration Data* tab. The calibration for each analyte can be viewed by clicking on the required isotope in the *Analyte* box. Each subsequent set of calibrations (calibration block) can be displayed by selecting the required calibration block from the drop-down combo box, e.g. *FQ Block 1*, *FQ Block 2*, etc. FQ denotes a Fully-Quantitative calibration and SQ denotes a Semi-Quantitative calibration, i.e. a response curve generated from the *FQ* calibrations. The SQ response curve is used to calculate semi-quantitative concentrations if required.

11.4.7. To view data, click on the *Numerical Results* tab. The *Analyte Dilution Conc.* tab is a tabular display of the calculated corrected concentrations for each analyte. These values have been corrected for internal standardization, external drift correction (if used), and dilution (where entered). The *Mass Uncorrected ICPS* tab shows the uncorrected raw data for each measured mass in units of integrated counts per second (ICPS). The *Analyte ICPS* tab shows integrated counts per second data that has been mathematically corrected for blank deduction, internal standardization, drift correction (if used), and dilution (as appropriate). The *Survey* tabs show the data integrated from the survey scan for each sample. Any concentrations displayed in the survey page will be semi-quantitative only.

11.4.8. To edit the amount of data on screen (filter the results display), click on the filter icon (funnel and lightening). Alter the numerical values or the check boxes to select the required data to display and click on *OK*. To jump directly to a particular sample of interest, find the sample in the drop-down combo box at the top of the data display and click on it.

11.4.9. To display mass-spectra, click on the *Spectra* tab. Display the spectrum for a particular sample by double-clicking on the sample name in the selection box on the left of the screen. Note that several spectra may be overlaid by double-clicking on each sample to be displayed. To zoom into a particular area, click the zoom icon (magnifying glass) and click and drag on the spectral display to zoom into the required area. The dashed-lines represent data acquired in the analogue mode of the detector whilst the solid-lines represent pulse-count data. To remove the noise associated with analogue detection at low signal levels, point at the display and right-mouse click to bring up a menu. Go to *View Options* and then click on *Eliminate Analogue Noise*. To identify a peak, click on it and wait for the options for that mass to be displayed in the box above the spectral display. To fingerprint a spectrum, double click on the species to fingerprint in the options box. This will overlay the isotopic pattern for the selected species, based on the lowest relative intensity signal for the pattern masses. The spectra may be navigated by using

the arrow buttons above the display. Allow the arrow cursor to hover over each button for an on-screen explanation of its function.

11.5. Post-Analysis Data Processing

11.5.1. Internal Standards

11.5.1.1. Check the internal standard recovery percentage for each internal standard isotope used for every sample. The percentage for each isotope must be within the range 30-120% for method 6020 and 60 – 125% for method 200.8.

11.5.1.2. If above 120%, check that the other internal standard isotopes show similar deviation. If not, this may be due to the presence of the internal standard element in the sample. This is particularly common with the isotopes of Li, Sc and Y in environmental materials. If this is the case, the affected internal standard isotope may be excluded for the sample affected, as follows. Go to the *Sample List*.

Find the sample affected and select it in the list by clicking on the box in the left-hand column. Click *Show Advanced* and go to *Internal Standards*. Click on *New Internal Standard Set*. Select the affected isotope(s) in the *Internal Standards* box on the right. Remove the affected isotope from the *Internal Standards* box by using the left hand arrow button (<<). Recalculate the results for this sample by going back to *Results* and clicking on *Refresh*.

11.5.1.3. If any internal standard isotope is outside the range 30-120% and all other internal standard isotopes show similar values for that sample, the instrument may have drifted, or the sample may be producing a suppression or enhancement effect. Find the nearest blank following the sample in question and check its internal standard results. If these are similarly reduced or elevated, the instrument has drifted and the samples must be reanalyzed from the last compliant blank. If the blank does not exhibit similar drift, the sample must be producing a suppression or enhancement effect due to its matrix. In this case the sample must be re-analyzed after a five-fold (1+4) or a ten-fold (1+9) dilution to reduce the matrix effect.

11.6. General protocols

11.6.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.6.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.6.3. An analytical run will consist of all customer samples and quality control samples analyzed under a daily initial calibration. Each new initial calibration will begin a new analytical run.

11.6.4. Type in the QC and sample information into the autosampler table.

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11.6.5. In order to use the ICP-MS data upload program into LIMS, the following naming conventions must be followed:

- Samples are identified by the 5-character work order number
- Matrix spikes, duplicates, and matrix spike duplicates are identified by the 5-character work order number followed by S (matrix spike), D (matrix spike duplicate) or X (sample duplicate).
- Prep Blanks are identified by the 5-character work order number followed by B.
- LCSs are identified by the 5-character work order number followed by C (LCS) or L (LCS Duplicate).

11.7. Initial Calibration

11.7.1. Open a new dataset using the date and instrument in the title. For instance the first run (A) on instrument 2 on JAN 1, 2003 would be X30101A.

11.7.2. Open the appropriate method if one already exists or create a new one for the analytes to be quantitated in the run. Solicit the assistance of a senior ICP-MS operator in creating a new method.

11.7.3. See Tables 7, 8, and 9 for recommended isotopes and interference equations for commonly analyzed elements.

11.7.4. If no recommended isotopes are given for the element to be analyzed, consult a senior ICP-MS operator or appropriate reference (see Section 13.2).

11.7.5. See Table 10 for commonly used internal standards.

11.7.6. All masses which could affect data quality should be monitored to determine potential interferences either simultaneously during an analytical run or in a separate scan.

11.7.7. Internal standards are added to all standards and samples by the instrument prior to analysis.

11.7.8. Use of an existing autosampler table is suggested. A read delay of 45 to 60 seconds is used between all analyses.

11.7.9. Calibration consists of a blank and a single calibration standard (STD1, see Table 2 for concentrations) in accordance with the manufacturer's procedure. Use the average of three integrations for both calibration and sample analyses.

11.8. The order of analysis for the initial QC samples and calibration should be:

1. Rinse
2. Performance Report (Tune Check)
3. STD1 (Calibration Standard)
4. STD2 (2x Calibration Standard)
5. STD3 (3X Calibration Standard)
6. ICV (Second source, must be ± 10% of true value)
7. ICB
8. CRI / RLV (Reporting Limit Verification Standard)

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9. ICSA (Interference check solution.)
10. ICSAB (Interference check solution, ± 20% of true value)
11. CCV
12. CCB
13. Prep QC such as LCS or MB, followed by samples (up to 10 runs)
14. Rinse
15. CCV
16. CCB

11.8.1. To continue the analytical run, add an additional 10 runs followed by a rinse and CCV/CCB, and repeat for up to 24 hours.

11.8.2. Analysis sequence when out-of-control QC is observed: Recalibrate and rerun all affected samples (including initial QC)

12. DATA ANALYSIS AND CALCULATIONS

12.1. All pertinent calculations are performed by the Plasma LAB software.

12.2. Reporting Requirements

12.2.1. Units are ug/L or mg/L for aqueous samples and mg/kg for soil samples and ug/wipe for wipe samples.

12.2.2. If dilutions were required due to insufficient sample, interferences, or other problems, the laboratory reporting limits are multiplied by the dilution factor.

12.2.3. For results less than 10, two significant figures will be reported. For results greater than or equal to 10, three significant figures will be reported. Refer to Policy QA-004 for additional information on significant figures and rounding.

12.2.4. Document any non-standard procedures or anomalies by using the anomaly program (Clouseau).

12.3. Data Package Requirements

12.3.1. A complete data package consists of: the daily tuning package, the method printout, run log, internal standard summary for 5.2 only, standards documentation, level 1 checklist, and all raw data.

12.3.2. Level I review will be completed by the analyst.

12.3.3. Level II review will be completed by a senior level laboratory analyst familiar with the technical aspects of ICP-MS and in accordance with the ICP-MS DATA REVIEW checklists. The instrument operator of an analytical run may not perform the Level II review for that run.

13. METHOD PERFORMANCE

13.1. Initial Demonstration of Capacity

Prior to analysis of any analyte using Method 6020, the following requirements must be met.

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13.2. Instrumentation Detection Limit (IDL) – IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined quarterly for method 6020 for the standard analytes listed in Appendix A. For method 200.8 IDLs will be determined annually. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined.

13.2.1. For 6020 the IDLs shall be determined by performing a blank analysis on 3 non-consecutive days with 7 consecutive measurements per day. The IDL is calculated by summing the standard deviations of the measurements from each day. For 200.8 the IDL is determined by performing 10 replicate blank analysis and mulitplying the resulting standard deviation by 3.

13.2.2. Each measurement must be performed as though it were a separate analytical sample.

13.2.3. Each measurement must be followed by a rinse and/or any other procedure normally performed between the analyses of separate samples.

13.2.4. The IDL measurement must consist of the same number of replicates used for analytical samples with the average result used for reporting.

13.2.5. **DoD samples cannot be analyzed without a valid IDL.**

13.2.6. **For DoD, the established IDL must be less than the MDL (see below) for each analyte.**

13.3. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. MDL's must be redetermined on an annual basis as detailed in Policy S-Q-003 and further defined in PITT-QA-007.

13.3.1. On occasion, a non-routine analyte is requested by the client. In lieu of a full MDL study, a standard containing the non-routine analyte must be analyzed. The concentration of the standard must correspond to the reporting limit or ½ the reporting limit. This is to verify that the method can satisfactorily quantify the element near the chosen reporting limit. The recovery of the standard must be between 50% and 150% of the expected value. The standard analysis should be kept with the analytical data.

13.4. Linear Range Verification (LR) - The linear range is determined semi annually (2x/year) for each element on the standard list. Some regulatory programs, such as AFCEE, may require more frequent determinations.

13.4.1. To determine the linear range, analyze 3 standards at increasing concentration up to 90% of the last concentration where the element was within 10% of true value is considered the upper linear range.

13.4.2. An alternative is to prepare a higher concentration standard and run this in the analytical run. If this standard is within 10% of the expected value this value can be used as the upper linear range. If this option is chosen, then note the action in an anomaly.

13.5. Training Qualification

13.5.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

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14. POLLUTION PREVENTION

14.1. 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."

15. WASTE MANAGEMENT

15.1. The following waste streams are produced when this method is carried out.

15.1.1. Acid waste consisting of sample and rinse solution. This waste is collected in waste containers identified as "Acid Waste", Waste #33. It is neutralized to a pH between 6 and 9 and then discharged down a lab sink.

15.1.2. Expired Metals Standards. This waste is collected in waste containers identified as "Acid Waste with Metals", Waste #6.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, September, 1994.

16.2. Thermo Electron X Series Users Manual

16.3. EPA Method 6020 CLP M, Version 8.

16.4. Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111), Method 200.8, Determination of Trace Elements in Waters by Inductively Coupled Plasma - Mass Spectrometry, Revision 5.4, 1994

16.5. EPA Method 200.8 EMSL Office of Research & Development, Cincinnati, OH (Draft Method, Revision 4.3, August 1990).

16.6. QA-003, TestAmerica Pittsburgh QC Program.

16.7. QA-004, Rounding and Significant Figures.

16.8. PITT-QA-DoD-0001, Implementation of the DoD QSM Version 3.

16.9. PITT-QA-007, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

Appendices

Appendix 1	Cleaning Procedure for Glass- and Plastic-ware
Appendix 2	Wash Solution Preparation Instructions
Appendix 3	Daily Instrument Maintenance
Appendix 4	Autotune and Performance Reports
Appendix 5	Resolution Setup
Appendix 6	Instrument Calibrations
Appendix 7	Sample Introduction Plumbing Diagram
Appendix 8	Procedure for Cleaning Sample Introduction Equipment and Cones
Appendix 9	Autosampler Position Map

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Appendix 10 ILM05.2D Contract Required Quantitation Limits (CRQLs)

Appendix 11 Spiking Levels

Appendix 12 Useful Web Links

Appendix 13 Work Flow Chart

Appendix 14 Glossary of Abbreviations

17.1. Tables

TABLE 1

STANDARD ANALYTE LIST AND REPORTING LIMITS*

Element	Symbol	CAS #	Aqueous RL mg/L	Aqueous QC SPIKE mg/L	Soil/Tissue RL mg/Kg	Soil/Tissue QC SPIKE mg/kg	Wipe RL ug/wipe	Wipe QC SPIKE ug/wipe
Aluminum	Al	7429-90-5	0.03	2.0	3.0	200	1.5	100
Antimony	Sb	7440-36-0	0.002	0.50	0.2	50	0.1	25
Arsenic	As	7440-38-2	0.002	0.04	0.2	4	0.05	100
Barium	Ba	7440-39-3	0.010	2.0	1.0	200	0.5	100
Beryllium	Be	7440-41-7	0.001	0.05	0.1	5	0.05	2.5
Boron	B	7440-42-8	0.005	1.0	0.5	100	0.25	50
Cadmium	Cd	7440-43-9	0.001	0.05	0.1	5	0.05	2.5
Calcium	Ca	7440-70-2	0.10	50	10.0	5000	5.0	2500
Chromium	Cr	7440-47-3	0.002	0.2	0.2	20	0.1	10
Cobalt	Co	7440-48-4	0.0005	0.5	0.05	50	0.025	25
Copper	Cu	7440-50-8	0.002	0.25	0.2	25	0.1	12.5
Iron	Fe	7439-89-6	0.05	1.0	5.0	100	2.5	50
Lead	Pb	7439-92-1	0.001	0.02	0.1	2	0.05	25
Magnesium	Mg	7439-95-4	0.10	50	10.0	5000	5.0	2500
Manganese	Mn	7439-96-5	0.0005	0.5	0.05	50	0.025	25
Molybdenum	Mo	7439-98-7	0.005	1.0	0.5	100	0.25	50
Nickel	Ni	7440-02-0	0.002	0.5	0.2	50	0.05	25
Potassium	K	7440-09-7	0.100	50	10.0	5000	5.0	2500
Selenium	Se	7782-49-2	0.005	0.01	0.5	1	0.25	100
Silver	Ag	7440-22-4	0.001	0.05	0.1	5	0.05	2.5
Sodium	Na	7440-23-5	0.10	50	10.0	5000	5.0	2500
Strontium	Sr	7440-24-6	0.005	1.0	0.5	100	0.25	50
Tin	Sn	7440-31-5	0.005	2.0	0.5	200	0.25	100
Titanium	Ti	7440-03-26	0.005	1.0	0.5	100	0.25	50
Thallium	Tl	7440-28-0	0.001	0.05	0.1	5	0.05	100
Vanadium	V	7440-62-2	0.001	0.5	0.1	50	0.05	25
Zinc	Zn	7440-66-6	0.005	0.5	0.5	50	0.25	25

* *Note: These are the routine reporting limits for most sample types. Lower reporting limits may be achievable for special projects. Difficult sample matrices may cause reporting limits to be raised.*

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TABLE 2			
Composition of the CAL Standard			
Element	**Concentration ug/mL**	**Element**	**Concentration ug/mL**
Ag	0.200	Mn	1.0
Al	1.00	Mo	0.200
As	0.200	Na	100
B	0.200	Ni	0.200
Ba	0.200	Pb	0.200
Be	0.200	Sb	0.200
Ca	100	Se	0.200
Cd	0.200	Si	10
Co	0.200	Sn	0.200
Cr	0.200	Sr	0.200
Cu	0.200	Ti	0.200
Fe	50	Tl	0.200
K	100	V	0.200
Mg	100	Zn	0.200

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TABLE 3
Composition of the ICV Standard

Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.08	Mn	0.4
Al	0.4	Mo	0.08
As	0.08	Na	40
B	0.08	Ni	0.08
Ba	0.08	Pb	0.08
Be	0.08	Sb	0.08
Ca	40	Se	0.08
Cd	0.08	Si	4.0
Co	0.08	Sn	0.08
Cr	0.08	Sr	0.08
Cu	0.08	Ti	0.08
Fe	20	Tl	0.08
K	40	V	0.08
Mg	40	Zn	0.08

TABLE 4
Composition of the ICSA Standard

Element	Concentration ug/mL	Element	Concentration ug/mL
Al	100	P	100
Ca	100	S	100
Fe	100	C	200
K	100	Cl^-	1000
Mg	100	Mo	2.0
Na	100	Ti	2.0

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TABLE 5
Composition of the ICSAB Standard

Element	Concentration ug/mL	Element	Concentration ug/mL
Ag	0.02	Na	100
Al	100	Ni	0.02
As	0.02	Pb	0.02
B	0.05	Sb	0.02
Ba	0.02	Se	0.05
Be	0.02	Si	0.50
Ca	100	Sn	0.10
Cd	0.02	Sr	0.02
Co	0.02	Ti	2.0
Cr	0.02	Tl	0.02
Cu	0.02	V	0.02
Fe	100	Zn	0.025
K	100	P	100
Mg	100.0	S	100
Mn	0.0225	C	200
Mo	2.00	Cl-	1000

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TABLE 6[1]

COMMON MOLECULAR ION INTERFERENCES IN ICP-MS

Molecular Ion	Mass	Element Interferences[2]	Molecular Ion	Mass	Element Interferences[2]
BACKGROUND MOLECULAR IONS					
NH^+	15		$^{38}ArH^+$	39	
OH^+	17		$^{40}ArH^+$	41	
OH_2^+	18		CO_2^+	44	
C_2^+	24		CO_2H^+	45	Sc
CN^+	26		ArC^+, ArO^+	52	Cr
CO^+	28		ArN^+	54	Cr
N_2^+	28		$ArNH^+$	55	Mn
N_2H^+	29		ArO^+	56	
NO^+	30		$ArOH^+$	57	
NOH^+	31		$^{40}Ar^{36}Ar^+$	76	Se
O_2^+	32		$^{40}Ar^{38}Ar^+$	78	Se
O_2H_+	33		$^{40}Ar_2^+$	80	Se
$^{36}ArH^+$	37				
MATRIX MOLECULAR IONS – Chloride					
$^{35}Cl0^+$	51	V	$^{37}Cl0H^+$	54	Cr
$^{35}Cl0H^+$	52	Cr	$^{35}Cl0^+$	51	V
$^{37}Cl0^+$	53	Cr	$^{35}Cl0H^+$	52	Cr
$Ar^{35}Cl^+$	75	As	$Ar^{37}Cl^+$	77	Se
MATRIX MOLECULAR IONS – Sulfate					
$^{32}SO^+$	48		$^{34}SOH^+$	51	V
$^{32}SOH^+$	49		SO_2^+, S_2^+	64	Zn
$^{34}SO^+$	50	V, Cr			
$Ar^{32}S^+$	72		$Ar^{34}S^+$	74	
MATRIX MOLECULAR IONS – Phosphate					
PO^+	47		PO_2^+	63	Cu
POH^+	48				
ArP^+	71				
MATRIX MOLECULAR IONS – Group I, II Metals					
$ArNa^+$	63	Cu	$ArCa^+$	80	
ArK^+	79				
MATRIX OXIDES[3]					
TiO	62-66	Ni, Cu, Zn	MoO	108-116	Cd
ZrO	106-112	Ag, Cd			

[1] *From Method 200.8, Section 13.2.6*
[2] *Method elements or internal standards affected by the molecular ions.*
[3] *Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes be monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.*

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TABLE 7

RECOMMENDED ANALYTICAL ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED [1]

Isotope	Element of Interest	Isotope	Element of Interest
27	Aluminum [2]	80,**78**,**82**,**76**,**77**,74	Selenium
121,123	Antimony [2]	**107**,**109**	Silver [2]
75	Arsenic [2]	**23**	Sodium [2]
138,**137**,136,**135**,134,132,130	Barium [2]	203, **205**	Thallium [2]
9	Beryllium [2]	**51**,**50**	Vanadium [2]
114,112,**111**,110,113,116,106,**108**	Cadmium [2]	**66**, **68**	Zinc [2]
42,**43**,**44**,46,48	Calcium [2]	83	Krypton
52,**53**,**50**,54	Chromium [2]	72	Germanium
59	Cobalt [2]	139	Lanthanum
63,**65**	Copper [2]	140	Cerium
56,**54**,**57**,58	Iron [2]	129	Xenon
206,207,**208**	Lead [2]	**118**	Tin
24,**25**,**26**	Magnesium [2]	105	Palladium
55	Manganese [2]	47,**49**	Titanium
98,96,92,97,94,95	Molybdenum	125	Tellurium
58,**60**,62,**61**,64	Nickel [2]	69	Gallium
39	Potassium [2]	35,37	Chlorine

[1] *From Method 6020 CLP-M, Table 9*

[2] *Element approved for ICP-MS determination by SW846 Method 6020 CLP-M*

NOTE: Isotopes recommended for analytical determination are **bolded**.

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TABLE 8

RECOMMENDED ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED

Rare Earth Elements	ICPMS Preferred Mass	Elemental Equations	Additional Masses
Lanthanum	138.906		
Cerium	139.905		
Praseodymium	140.907		
Neodymium	141.908	-0.125266 * ^{140}Ce	142.910, 144.912
Samarium	151.920	-0.012780 * ^{157}Gd	144.912
Europium	152.929		
Gadolinium	157.924	-0.004016 * ^{163}Dy	156.934
Terbium	158.925		
Dysprosium	163.929	-0.047917 * ^{166}Er	
Holmium	164.930		
Erbium	165.930		
Thulium	168.934		
Ytterbium	173.939	-0.005935 * ^{178}Hf	171.937
Lutetium	174.941		

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TABLE 8			
RECOMMENDED ISOTOPES AND ADDITIONAL MASSES WHICH MAY BE MONITORED			
Rare Earth Elements			
Other Elements			
Boron	11.009		
Calcium	43.956		
Cesium	132.905		
Galium	68.926		
Germanium	71.922		
Gold	196.967		
Hafnium	177.944		176.944
Holmium	164.930		
Iridium	192.963		
Lithium	7.016		
Tungsten	183.951	-0001242* ^{189}Os	
Uranium	238.050		
Yttrium	88.905		
Zirconium	238.050		
Niobium	92.906		
Palladium	104.905		
Phosphorus	30.994		
Platinum	194.965		
Rhenium	186.965	-0.099379 * ^{189}Os	
Rhodium	102.905		
Rubidium	84.912		
Ruthenium	101.904	-0.045678 * ^{105}Pd	
Scandium	44.956		
Strontium	87.906		
Tantalum	180.948		
Tellurium	127.905	-0.072348 * ^{129}Xe	
Thorium	232.03		

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TABLE 9

ELEMENTAL EQUATIONS USED TO CALCULATE RESULTS

Element	Elemental Equation	Note
Al	(1.000) (^{27}C)	
Sb	(1.000) (^{121}C)	
As	(1.000) (^{75}C) - (3.1278)[^{77}C) - (1.0177)(^{78}C)]	Correction for chloride interference with adjustment for Se77. ArCl 75/77 ratio may be determined from the reagent blank.
Ba	(1.000) (^{137}C)	
Be	(1.000) (^{9}C)	
Cd	(1.000) (^{111}C) - (1.073) [(^{108}C) - (0.712) (^{106}C)]	Correction of MoO interference. An additional isobaric elemental correction should be made if palladium is present.
Cr	(1.000) (^{52}C)	In 0.4% v/v HCl, the background from ClOH will normally be small. However the contribution may be estimated from the reagent blank.
Co	(1.000) (^{59}C)	
Cu	(1.000) (^{63}C)	
Pb	(1.000) (^{206}C) + (1.000) (^{207}C) + (1.000) (^{208}C)	Allowance for isotopic variability of lead isotopes.
Mn	(1.000) (^{55}C)	
Mo	(1.000) (^{98}C) - (0.146) (^{99}C)	Isobaric elemental correction for ruthenium.
Ni	(1.000) (^{60}C)	
Se	(1.000) (^{82}C)	Some argon supplies contain krypton as an impurity. Selenium is corrected for Kr82 by background subtraction.
Ag	(1.000) (^{107}C)	
Tl	(1.000) (^{205}C)	
Th	(1.000) (^{232}C)	
U	(1.000) (^{238}C)	
V	(1.000) (^{51}C) - (3.127) [(^{53}C) - (0.113) (^{52}C)]	Correction of chloride inference with adjustment for Cr53. Cl0 51/53 ratio may be determined from the reagent blank.
Zn	(1.000) (^{66}C)	
Internal Standards		
Bi	(1.000) (^{209}C)	
In	(1.000) (^{115}C) -(0.0149) (^{118}C)	Isobaric elemental correction for tin.
Ge	(1.000) (^{72}C)	
Sc	(1.000) (^{45}C)	
Tb	(1.000) (^{159}C)	
Tm	(1.000) (^{169}C)	
Y	(1.000) (^{89}C)	

* Method elements or internal standards affected by the molecular ions.

C = Calibration blank subtracted counts at specified mass.

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TABLE 10		
INTERNAL STANDARDS AND LIMITATIONS OF USE		
Internal Standard	Mass	Possible Limitation
Lithium	6	a
Scandium	45	Polyatomic Ion Interference
Germanium	72	
Yttrium	89	a, b
Rhodium	103	
Indium	115	Isobaric Interference by Sn
Terbium	159	
Holmium	165	
Thulium	169	
Lutetium	175	
Bismuth	209	a

a May be present in environmental samples.

b In some instruments Yttrium may form measurable amounts of YO^+ (105 amu) and YOH^+ (106 amu). If this is the case, care should be taken in the use of the cadmium elemental correction equation.

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Appendices

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Appendix 1

Cleaning Procedure for Glass- and Plastic-ware

All glassware and plastic-ware coming into contact with samples, reagents and standards must be cleaned in the following manner. Plastic pipette tips may be cleaned in the same manner by soaking them in a suitable plastic container.

1) Completely fill the container to be leached with 10% nitric acid solution (6.1.5) and fit the lid.
2) Leave soaking for at least 12 hours.
3) Empty the container of acid and rinse thoroughly with laboratory water (6.1.1). Note that the acid may be collected and re-used until it becomes too contaminated.
4) Allow the vessel to air-dry in a clean area (preferably Class-1000 or better). If no such clean area is available, the container should be allowed to dry in the cleanest possible environment, or may be emptied of residual water as much as is possible and re-capped.
5) Containers should be capped ready for use and stored in the cleanest area available.
6) If pre-cleaned containers are to be stored for long periods (weeks to months) prior to use, it is most effective to store them full of laboratory water (6.1.1). This must be discarded and the containers rinsed thoroughly with laboratory water (6.1.1) and dried before use.

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Appendix 2

Wash Solution Preparation Instructions (2% Nitric Acid (v/v))

A large volume of this solution is required for supply to the autosampler rinse station in order to wash the probe between samples. These instructions detail the preparation procedure for 2.5 L of this solution that is normally sufficient for one day of analytical use. The procedure may be scaled up or down as required.

1) Into a 2.5 L container (pre-cleaned as per Appendix 1), add 500±450 mL of laboratory water (6.1.1)
2) Add 50±10 mL of concentrated nitric acid (6.1.3)
3) Make to 2.50±0.25 L with laboratory water (6.1.1)
4) Mix well

Notes:

If preparing larger quantities simply scale-up quantities proportionally.

If analyzing for Ag, add hydrochloric acid at 1% by adding 50±10 mL of concentrated hydrochloric acid (6.1.2) after step 2.

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Appendix 3

Daily Instrument Maintenance

1) Wipe all instrument, autosampler and surrounding bench surfaces with a damp wipe – continual cleanliness is important for the minimization of contamination
2) Check Wash Solution volume and remake if necessary (see Appendix 2)
3) Empty Waste Vessel according to laboratory disposal policy
4) Check the condition of all peristaltic pump tubes and replace if required (it is recommended to replace these daily although this may not be necessary with lower sample loads)
5) Check condition of sample introduction system and cones and clean and/or replace as necessary (see Appendix 8)
6) Ensure instrument fume-extraction system is operational

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Appendix 4
Autotune and Performance Reports

Description

Autotune is a *PlasmaLab* software tool that allows the X Series to be optimized in a consistent, routine manner, giving reproducible levels of performance and saving the operator time and effort. It works by following a pre-defined sequence, optimizing individual instrument parameters in turn. Default sequences are provided with the software upon installation and a further customized sequence is provided on the CD accompanying this productivity pack.

Performance Reports are a *PlasmaLab* software tool that allows the X Series performance to be checked on a daily basis. The *Performance Report* can be set-up to give information about instrument sensitivity, stability, background, oxide species, doubly charged species, mass-calibration validity and peak resolution. Like *Autotune*, the *Performance Report* is user definable but defaults are provided with the software. Customized *Performance Reports* are provided on the CD accompanying this package.

The philosophy of use of these tools is as follows. After the sample introduction system or the cones have been removed and replaced or upon using the instrument for the first time or following major adjustments, the full *Autotune* sequence should be used to properly optimize the system. This takes about 15 minutes. From this, an *Autotune Update* sequence can be automatically created. This is a shortened version of the optimization sequence and will take about 5 minutes to run. The performance of the X Series is, in general, very stable from day-to-day, meaning that large amounts of optimization are not normally needed on a daily basis. To check whether optimization is needed, a *Performance Report* can be run initially. The results of this tell the operator if the system requires resolution adjustment, re-mass-calibration, or re-optimization. If the required sensitivity, background, stability or oxide performance is not satisfied, an *Autotune* should be run (the faster *Autotune Update* is normally sufficient). The *Performance Report* should then be repeated to ensure that the problem has been resolved.

Installing the EPA Autotune Sequence

To install the custom Autotune sequence, follow the instructions below:

1) Insert the CD in the CD ROM drive of the instrument operating PC. Wait for it to autorun and install the Productivity Pack by following the prompts after clicking on *Install*.
2) Ensure that PlasmaLab version 2.2 (or higher) has been installed

3) In PlasmaLab, go to *Instrument*, *Tune* and click on the down arrow button next to the *Autotune* icon (musical note).
4) Point to *Tools* in the menu and then select *Import Autotune Sequences*
5) Click *Next* in the Autotune Wizard
6) Click on *Browse* and find the path

C:/Program Files/ThermoElemental/PlasmaLab/Data

7) Select *EPA Autotune Sequence* and click on *Open*
8) Click on *Next*
9) Select *EPA – Xi Interface* and click on *Next*
10) Click on *Finish*

Installing the EPA Performance Reports

To install the custom Performance Reports, follow the instructions below:

1) Ensure the Pack is installed from the CD as described above
2) Ensure that PlasmaLab version 2.2 (or higher) has been installed
3) In PlasmaLab, go to *Instrument*, *Tune* and click on the down arrow button next to the *Performance Report* icon (musical note on page).
4) Point to *Tools* in the menu and then select *Import Performance Report*
5) Click *Next* in the Performance Report Wizard
6) Click on *Browse* and find the path for the CD ROM drive

   C:/Program Files/ThermoElemental/PlasmaLab/Data
7) Select *EPA 6020 Report* and click on *Open*
8) Click on *Next*
9) Select *EPA 6020 2.1* and click on *Next*
10) Click on *Finish*

To install the second Performance Report, follow instructions 1) to 10) above, selecting the alternative Performance Report name, i.e. *EPA ILM05_2D Report*.

Running Autotune from the Tune Page

To run an Autotune Sequence, follow the instructions below:

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1) In PlasmaLab go to *Instrument*, *Tune* and click on the *Autotune* icon (musical note)
2) Select *Run an Existing Autotune Sequence* and click on *Next*
3) Select the required sequence, e.g. *EPA Xi Interface*, or *EPA Xi Interface – Update* and click on *Next*
4) Ensure that the indicated solution is being aspirated (through both probes if on-line internal standard addition is being used) and allow sufficient time for the solution to be transported into the nebulizer
5) Click on *Finish*

The selected Autotune sequence will now be run. To monitor its progress, observe the processes indicated at the bottom left of the PlasmaLab screen and open the Service Window (double-click on *MS* icon at the bottom right of the screen). A printable *Autotune Report* is generated at the end of the sequence. To continue, this report must be closed. To access this report upon closure, go to *Instrument*, *Configurations*, *Configuration Editor* and point to the appropriate *Instrument Settings* line. Open a pop-up menu by right-clicking and use the *View Tune Report* selection.

Running a Performance Report from the Tune Page

To run a Performance Report, follow the instructions below:

1) In PlasmaLab go to *Instrument*, *Tune* and click on the *Performance Report* icon (musical note on a page)

2) Select *Run an Existing Performance Report* and click on *Next*

3) Select the required sequence, e.g. *EPA ILM05 / 6020*, or *EPA 6020* and click on *Next*

4) Ensure that the indicated solution is being aspirated (through both probes if on-line internal standard addition is being used) and allow sufficient time for the solution to be transported into the nebulizer

5) Click on *Finish*

   The selected *Performance Report* will now be run. To monitor its progress, open the Service Window (double-click on *MS* icon at the bottom right of the screen). A printable *Performance Report* is generated at the end of the sequence. To access this report upon closure, go to *Instrument*, *Tune*, and click on the down arrow to the right of the Performance Report icon. Point at *Tools* and then select *View Performance Report Results*. Select the required Performance Report to view and click *OK*.

Running Performance Reports and Autotune in an Experiment

It is also possible to automate the running of these procedures using an instrument setup sample within an experiment. To do this, insert an *Instrument Setup Sample* at the beginning of the Sample List by selecting the first sample and using a right-mouse-click menu to *Insert New Before*. Define the *Sample Type* for this new sample as *Instrument Setup* and click on *Show Advanced*. Click on the *Instrument Performance Tests* tab and setup the Performance Report and Autotune functions following the logic and using the drop-down combo boxes to select the next action. An example would be as follows:

Acquire Performance Report	*EPA ILM05.2 / 6020*
If mass calibration verification fails then	*Abort the Queue*
If the Performance Report fails then	*Autotune using EPA – Xi Interface*
If the Autotune fails then	*Abort the experiment*
If the Autotune passes then	*re-run the Performance Report*
If the Performance Report fails again then	*Abort the Queue*

When Performance Reports and Autotunes are acquired in this way, the results are stored as part of the experiment report. Note that since this method of acquiring the report is done using the autosampler, the solution concentration should be adjusted if on-line internal standard addition is to be used, e.g. if the addition dilutes the samples 1:1, the solution concentration should be doubled to get an accurate measure of sensitivity.

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Appendix 5
Resolution Set up

With the instrument in *Operate* mode, aspirate 10-µg/L Tune solution (6.4.1) (through both probes if using on-line internal standard addition). Go to *Instrument*, *Tune* and stop the real time display (RTD) using the square stop icon. Change the display mode from *Time vs ICPS* to *ICPS on the full mass range*. Insert Be as the mass to monitor and change the spacing to 10, the dwell to 1 ms and the channels to 200. Disable all other masses in the grid. Restart the RTD by clicking on the triangular play icon. The software will display the scanned peak for mass 9, Be. To adjust the resolution, go to the *Global* tab and use the slider bar marked *Standard resolution*. This must be set up to give a peak width of less than 0.75 amu at 5% peak height. This is typically reached at a setting of between 100 and 200. If high-resolution mode is to be used, this can be setup by changing the resolution setting on the RTD to *High*. The High Resolution peak width is typically set at about 0.4 amu at 5% peak height, again with values typically between 100 and 200. Note that this method does not use High-resolution mode. Each resolution mode should be checked with several other masses across the mass range, typically 55Mn, 115In, 203Tl and 238U are used. Special attention should be paid to the resolution setup for Mn. This is measured at m/z 55, which is adjacent to both iron and argon oxide at mass 56. These high signals must be properly resolved from the low Mn signal in standard resolution mode. When the correct resolution settings are achieved, save the setting using the disk icon. **Note that a new mass-calibration must always be performed after adjustment of the resolution.**

17.2. Appendix 6

17.3. Instrument Calibrations

There are three instrument calibrations that are fundamental for obtaining good quality data on the X Series. These are:

1) Mass-calibration
2) Detector Plateau and Analogue voltage set routines
3) Detector cross-calibration.

Mass calibration sets the quadrupole scan parameters to give the correct measured mass positions. The detector plateau sets the optimum voltage on the ion or pulse counting section of the discrete dynode detector. The analogue voltage set routine applies an appropriate voltage on the analogue part of the detector to obtain a cross-calibration factor of approximately 20,000 for a mid-mass isotope. The detector calibration, or cross-calibration, calculates the correction factor, for each measured mass, between the two detector modes, pulse counting and analogue. All three calibrations may be performed in a single routine, or may be performed separately.

Mass Calibration

A mass-calibration must be performed whenever the resolution settings are adjusted, as this will affect the apparent mass position. Mass-calibration must be performed when the Performance Report shows that measured peak positions are >0.1 amu from their nominal position. Mass-calibrations are best performed using a solution containing as many elements as possible or with every analyte required for analysis at the very least. The solution should contain Li and U as these are used as low and high mass datum points. An appropriate concentration solution be used (one that gives between **100,000-1,500,000 cps** for each mass to be calibrated is appropriate). To perform a mass calibration, follow the instructions below.

1) Click *Experiment*
2) Select *Create New Experiment*
3) Click *OK*
4) Select the *Default* database
5) Click *Open*
6) Go to *Sample List*
7) Click the *Report* check box in the sample list grid
8) Use the drop-down combo box in the *Type* column to select *Instrument Setup*
9) Click on the *Show Advanced* button

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10) Click on the *Instrument Calibrations* tab

11) Check the *Mass-Calibration* box

12) There is an option to *Update current mass-calibration* or form a *New mass-calibration*. Unless a major hardware change has been performed, the *Update current mass-calibration* option should be selected.

13) Click *Queue*

14) Save the experiment with an appropriate name, e.g. *masscal 090902* and click *Save*

15) Click *Append*

16) Click *OK*

Mass-calibration will now be performed.

To view the mass-calibration results, go to *Instrument*, *Calibrations*, *Mass-Calibration*. A mass-calibration for each of the two resolution modes is displayed in the graph of Peak Width and Error (y) versus Mass (x). The current mass-calibration is indicated by the row(s) displayed in green. To display alternative mass-calibrations, click on the appropriate date/time-stamped line in the top grid. The Performance Report function can be used to check mass-calibration accuracy (see Appendix 4).

Detector Plateau and Analogue Voltage Set

These routines can be performed separately, but it is advised to run them simultaneously as described here. The necessary frequency of these calibrations depends upon the amount of signal the detector is exposed to, i.e. how many samples are analyzed, which analytes and what concentrations. For most laboratories running a moderate sample load, this procedure may be run weekly. Up to three masses may be used in this procedure, however here, the use of a single mass is described. A solution that gives a countrate of between **100,000-1,500,000 cps** is appropriate. The default mass used here is indium (m/z 115), so this must be present in the solution for the routine to work. For an X5 instrument, an appropriate concentration would typically be between 10 and 100 µg/L, depending upon the sensitivity of the system. To perform this routine, follow the instructions below.

1) Click *Experiment*

2) Select *Create New Experiment*

3) Click *OK*

4) Select the *Default* database

5) Click *Open*

6) Go to *Sample List*

7) Click in the *Report* check box in the sample list grid

8) Use the drop-down combo box in the *Type* column to select *Instrument Setup*

9) Click on the *Show Advanced* button

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10)Click on the *Instrument Calibrations* tab

11)Check *the Set analogue voltage* box

12)Set the *Number of iterations* to *2*

13)Click *Queue*

14)Save the experiment with an appropriate name, e.g. *plateau 090902* and click *Save*

15)Click *Append*

16)Click *OK*

The voltage setup will now be performed. To view the plateau, go to *Instrument*, *Calibrations*, *Detector Plateau*. A graph of signal intensity (y) versus voltage (x) is displayed. The “knee” inflexion on this plot corresponds to the plateau voltage. This is automatically selected and applied to the detector by the software.

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Detector Calibration (Cross-Calibration)

This routine must be performed whenever the detector voltages are altered and daily prior to analysis of samples. The solution used must contain all the analytes to be measured as an absolute minimum. The more analytes present, the better. All analytes should ideally be set at a concentration that gives between **500,000 and 1,500,000cps**. To perform the detector calibration, follow the instructions below:

1) Click *Experiment*
2) Select *Create New Experiment*
3) Click *OK*
4) Select the *Default* database
5) Click *Open*
6) Go to *Sample List*
7) Click in the *Report* check box in the sample list grid
8) Use the drop-down combo box in the *Type* column to select *Instrument Setup*
9) Click on the *Show Advanced* button
10) Click on the *Instrument Calibrations* tab
11) Check the *Detector Calibrate* box
12) Click *Queue*
13) Save the experiment with an appropriate name, e.g. *xcal 090902* and click *Save*
14) Click *Append*
15) Click *OK*

The detector calibration will now be performed. To view the cross-calibration grap, go to *Instrument*, *Calibrations*, *Detector Cross-Calibration*. A graph of cross-calibration factor (y) versus mass (x) is displayed. **Use the data table to check that all analytical masses of interest have been used in the cross-calibration**. If not, the cross-calibration factor will be estimated from the equation of the graph. This may result in error.

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All Routines in One

It is possible to run all three of the above routines on a single run if the solution used conforms to all of the criteria spelt out above. To do this, follow the instructions below.

1) Click *Experiment*
2) Select *Create New Experiment*
3) Click *OK*
4) Select the *Default* database
5) Click *Open*
6) Go to *Sample List*
7) Click in the *Report* check box in the sample list grid
8) Use the drop-down combo box in the *Type* column to select *Instrument Setup*
9) Click on the *Show Advanced* button
10) Click on the *Instrument Calibrations* tab
11) Check the *Mass calibration*, *Detector Calibrate* and *Set analogue voltage* boxes
12) Set the *Number of iterations* to *2*
13) Click *Queue*
14) Save the experiment with an appropriate name, e.g. *instr cal 090902* and click *Save*
15) Click *Append*
16) Click *OK*

The instrument calibrations will now be performed. Each parameter can be viewed as described above.

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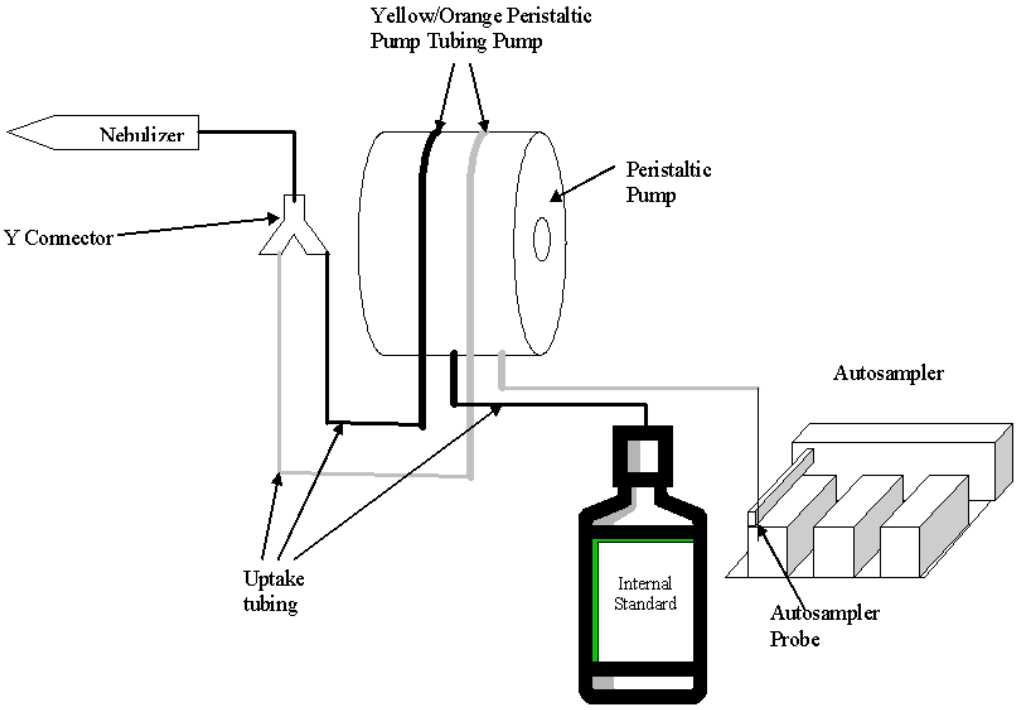
Appendix 7

Sample Introduction Plumbing Diagram



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Appendix 8

Procedure for Cleaning Sample Introduction Equipment and Cones

1) Ensure that the instrument is in the *vacuum* or *shutdown* state (i.e. the plasma is OFF and the slide valve is SHUT)
2) Dismantle the sample introduction system as follows:

a) Remove the gas connection from the nebulizer

b) Remove the sample input plug from the nebulizer

c) Remove the metal clip on the spray chamber to elbow joint

d) Remove the drain plug from the spray chamber

e) Slide the spray chamber and nebulizer away from the elbow

f) Carefully slide the nebulizer out of the spray chamber and set both pieces aside in a safe place

g) Open the torch box and the internal Faraday cage

h) Pull the gas connections away from the torch

i) Undo the torch catch

j) Remove the metal clip on the elbow to torch joint

k) Carefully remove the torch from the load coil and set aside in a safe place

l) Remove the elbow by sliding it out of the torch box bulkhead toward spray chamber end

m) Slide the torch box away from the mass spectrometer to reveal the interface

n) Use the flat metal cone tool to undo the locking ring over the sample cone

o) Carefully remove the sample cone and set aside in a safe place

p) Carefully unscrew and remove the skimmer cone from the interface using the cylindrical aluminium tool and set aside in a safe place

3) Clean the cones as follows.

a) Carefully place the cones into a large beaker and fill with sufficient 0.05% nitric acid to cover – CAUTION: Stronger acids will corrode the cone material and reduce lifetime

b) Place the beaker in an ultrasonic bath for about 10 minutes or until surface deposition has been removed

c) Carefully remove the cones from the solution and rinse thoroughly with deionised water

d) Allow the cones to air-dry prior to refitting

4) Clean the sample introduction equipment as follows.

e) Carefully place the glass sample introduction components into a large beaker and fill with sufficient 10% nitric acid to cover all components

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f) Place in an ultrasonic bath for between 20 minutes and 1 hour

g) Carefully remove the glass components and rinse thoroughly with deionised water

h) Allow to air-dry prior to refitting

5) Reassemble the components in the reverse order to disassembly

Note: Occasionally, glass sample introduction components crack when the ultrasonic cleaning procedure is used. To avoid this, the components may be soaked in acid, as above, for 12 hours, without ultrasonic treatment.

Thermo Electron cannot take any responsibility for any breakage that occurs during cleaning.

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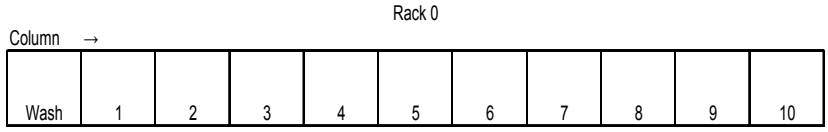
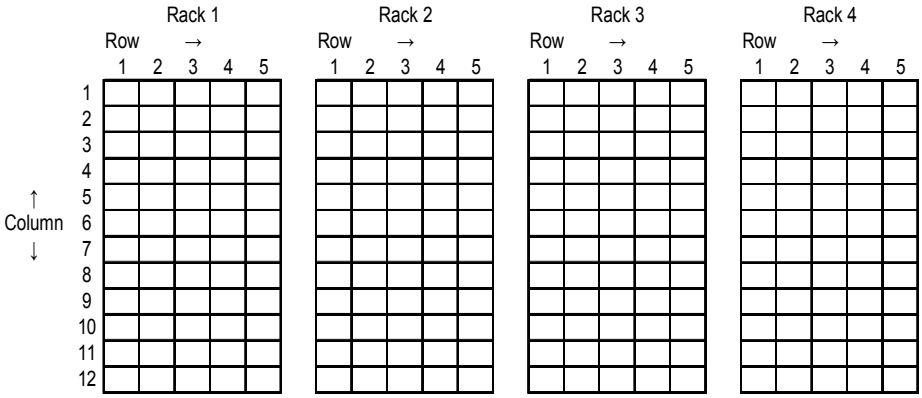
Appendix 9

Autosampler Position Map





NB: This map is only applicable for CETAC ASX-500/510 autosamplers fitted with 60 position racks.

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Appendix 10

ILM05.2D Contract Required Quantitation Limits (CRQLs)

Analyte	CRQL (µg/L)
Al	30
Sb	2
As	1
Ba	10
Be	1
Cd	1
Ca	(100)
Cr	2
Co	0.5
Cu	2
Fe	(50)
Pb	1
Mg	(100)
Mn	0.5
Ni	1
K	(100)
Se	5
Ag	1
Na	(100)
Tl	1
V	1
Zn	2

CRQLs given in parentheses are not specified for ICP-MS in EPA document ILM05.2 and are for ICP-AES. This is for information only.

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Appendix 11

Spiking Levels

(Concentration in Final Solution Based on Instructions Within this Document)

Analyte	Spike Value (µg/L)
Al	2000
Sb	500
As	40
Ba	2000
Be	50
Cd	50
Cr	200
Co	500
Cu	250
Pb	20
Mn	500
Ni	500
Se	10
Ag	50
Tl	50
V	500
Zn	500

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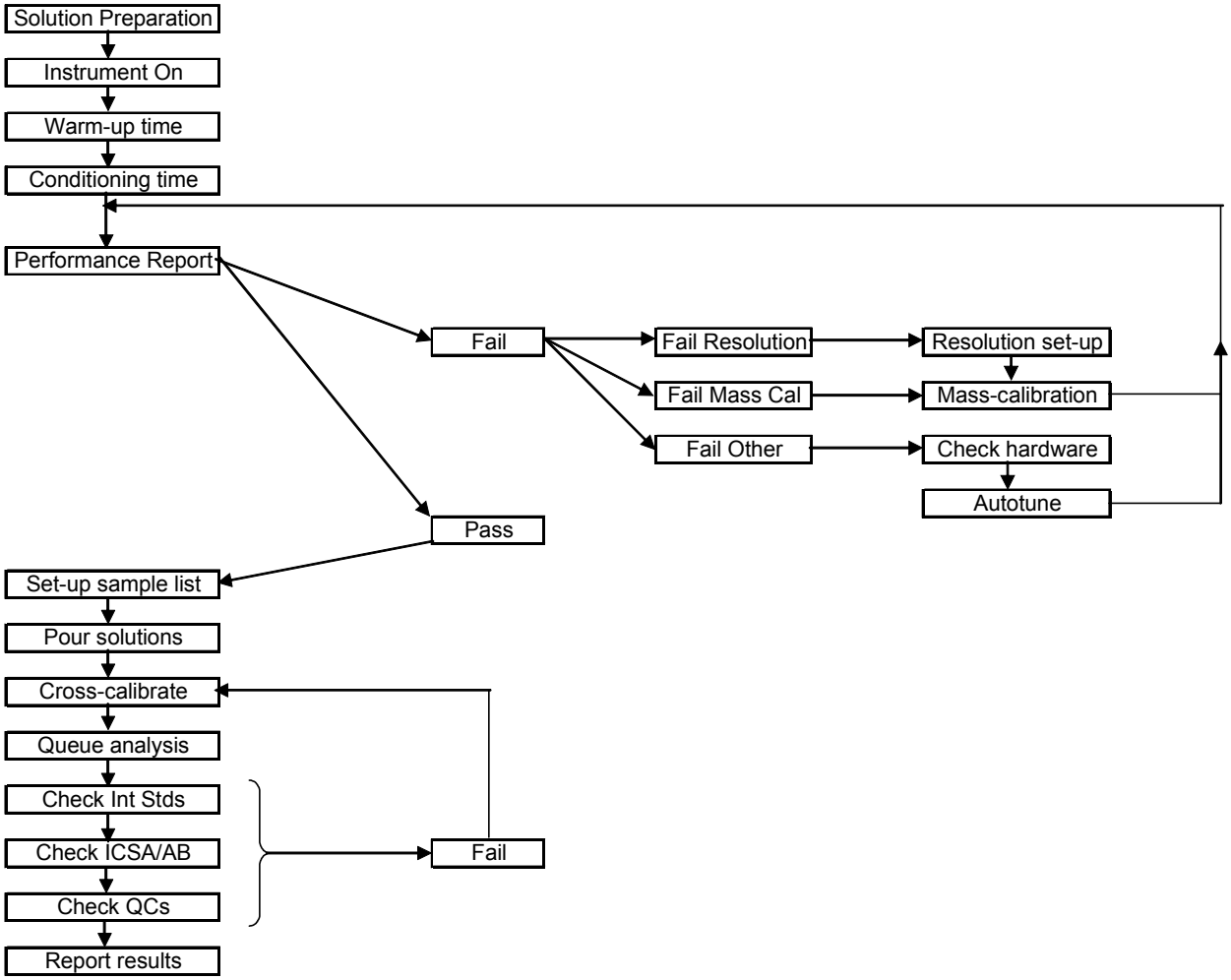
Appendix 12
Work Flow-Chart



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Appendix 13

Glossary of Abbreviations

QC Code	QC Name	Purpose	Frequency	Limits
ICV	Initial Calibration Verification	checks the calibration against a second calibration source	After initial calibration	90-110%
ICB	Initial Calibration Blank	initial check of read-back at blank level	After initial calibration	<CRQL (1)
CRI	Contract Required Quantitation Limit Check	checks accuracy at the required limit of quantitation	After each calibration and every 20 samples	50-150% (1)
ICSA	Interference Check Solution A	checks for freedom from interference	After initial calibration	±3CRQL or ±20% of the true value (whichever is the greater) (1)
ICSAB	Interference Check Solution AB	checks that analytes are accurately measured in an interference-producing matrix	After initial calibration	80-120% of true value
CCV	Continuing Calibration Verification	a continuing periodic check on accuracy and drift	After each calibration and every 10 samples	90-110%
CCB	Continuing Calibration Blank	a continuing periodic check on the read-back at blank levels	After each calibration and every 10 samples	<CRQL (1)

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QC Code	QC Name	Purpose	Frequency	Limits
PDS	Post Digestion Spike	checks the recovery of analytes spiked into an unknown sample after preparation (digestion)	Once every 20 samples per matrix	75-125%
DUP	Duplicate	checks the reproducibility of results by analyzing an unknown sample in duplicate	Once every 20 samples per matrix	±20% Relative Percentage Difference (RPD)
SER	Serial Dilution	checks for matrix effects by assessing the variation of results for an unknown sample before and after dilution	Once every 20 samples per matrix	±10% of the original undiluted result after dilution correction
LCS	Laboratory Control Sample	checks the accuracy of the entire analytical process	Once every 20 samples per matrix	80-120%

[(1)] For specific DoD requirements, refer to PITT-QA-DoD-0001.

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STL

LABORATORY-SPECIFIC

STL STANDARD OPERATING PROCEDURE

TITLE: EXTRACTABLE RESIDUE (LIPIDS) FROM ANIMAL TISSUE

(SUPERSEDES: REVISION 2)

Prepared by:	Erica M Bozik	5/1/07 Date
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1. SCOPE AND APPLICATION

1.1. This SOP describes the procedure for determining organic solvent extractable residue from fish tissue. Normally this residue is predominantly lipid material from the tissue, but it may include other non-polar material as well (e.g. petroleum hydrocarbons).

This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above.

2. SUMMARY OF METHOD

2.1. A 10-gram aliquot of homogenized tissue is extracted via soxtherm. The extract is dryed and evaporated to dryness. The residue remaining after evaporation is determined gravimetrically.

3. DEFINITIONS

3.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP), current version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, Lab Specific Addendum to the CSM, and this document.

5.2. 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.

5.3. Ultrasonic disrupters can produce high intensity noise and must be used in an area with adequate noise protection.

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5.4. 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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.6. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.7. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a

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laboratory supervisor and/or the EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Syringe or positive displacement pipette: 1 mL

6.2. Analytical balance, capable of accurately weighing ± 0.0001 g

6.3. Toploader Balance: >100 g capacity, accurate ±0.1 g

6.4. Soxtherm Model S 306A

6.5. Soxtherm thimbles

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methylene chloride, pesticide grade or equivalent

7.1.2. Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.

7.1.3. Fish Oil (Sigma): purchased commercially.

7.2. Standards

7.2.1. Not applicable

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The tissue samples are stored frozen and are to be extracted within 1 year of sample collection.

8.2. The extracts are stored at ambient temperature and analyzed within forty (40) days of extraction.

9. QUALITY CONTROL

9.1. Batch Definition

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9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, LCSD, Method Blank), which are processed similarly, with respect to the procedure. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. Corrective Action for Blanks

9.2.2.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.2.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD): A LCS and LCSD must be processed with each batch of 20 or less samples. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.1. Corrective Action for LCS

9.3.1.1. The LCS/LCSD recoveries must be evaluated against in-house control limits. If the results are outside established control limits, the system is out of control and corrective action must occur.

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9.3.1.2. Corrective action will include repreparation and reanalysis of the batch unless the client agrees that another corrective action is acceptable. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative**.

9.4. Surrogates

9.4.1. Not applicable.

9.5. Duplicates

9.5.1. Sample duplicates are performed at a frequency of one per analytical batch of up to 20 samples.

9.6. Nonconformance and Corrective Action

9.6.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Not Applicable

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Procedure

11.3.1. Weigh 10 g of the homogenized tissue into a soxtherm thimble. Record the weight to the nearest 0. 1 g on the benchsheet. Add sodium sulfate until no free liquid is present in sample. The method blank consists of 10 grams of sodium sulfate. The

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LCS/LCSD consist of 10 grams of sodium sulfate and spiked with 1 g of Fish Oil (Section 7.1.3).

11.3.2. Add 120 mL of methylene chloride.

11.3.3. Extract at 150°C.

11.3.4. Remove from soxtherm and cool.

11.3.5. Transfer to a pre-weighed 40-mL VOA vial.

11.3.6. Concentrate to dryness.

11.3.7. Re-weigh dried VOA vial.

11.3.8. Proceed to Section 12.

11.4. Analytical Documentation

11.4.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, and any corrective actions or modifications to the method.

11.4.2. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.4.3. Sample results and associated QC are entered into the LIMS after final technical review.

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12. DATA ANALYSIS AND CALCULATIONS

$$Concentration(\%) = \frac{(A - B)}{W} \times 100$$

Where:

A = Weight of beaker + residue, g
B = Weight of the beaker, g
W = Weight of sample extracted, g

13. METHOD PERFORMANCE

13.1. Training Qualifications:

13.1.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use, and has the required experience.

14. POLLUTION PREVENTION

14.1. 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. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. The following waste streams are produced when this method is carried out.

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15.1.1. Extracted solid samples contaminated with methylene chloride. This waste is collected in waste container identified as "Lab Trash Waste", Waste #12.

15.1.2. Used sodium sulfate contaminated with methylene chloride from the extract drying step. This waste is collected in waste container identified as "Lab Trash Waste", Waste #12.

15.1.3. Assorted flammable solvent waste from various glassware rinses. This waste is collected in waste containers identified as "Mix Flammable Solvent Waste", Waste #3.

15.1.4. Methylene chloride waste from various glassware rinses. This waste is collected in waste containers identified as "Methylene Chloride Waste", Waste #2.

15.1.5. Miscellaneous disposable glassware contaminated with solvents and sample residue. This waste is collected in waste container identified as "Lab Trash Waste", Waste #12.

16. REFERENCES

16.1. References

16.1.1. STL Quality Assurance Management Plan (QAMP), current version.

16.1.2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Method 8290 Sections 6.7, 7.2.2, 7.3.3, and Method 3550.

16.1.3. United States Army Corps of Engineers Waterways Experiment Station. May 1995. A Comparison of Three Lipid Extraction Methods. Technical Note EEDP-01-35. 3909 Halls Ferry Road, Vicksburg, Mississippi 39180-6199.

16.2. Associated SOPs

16.2.1. PITT-OP-0001, Extraction and Cleanup of Organic Compounds from Waters and Soils, SW846 3500-series and 3600-series, and EPA 600-series methods, current version.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1.1. Not applicable.

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TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

PITTSBURGH LABORATORY STANDARD OPERATING PROCEDURE

TITLE: SAMPLE RECEIVING AND CHAIN OF CUSTODY

(SUPERSEDES: SOP NO.: PITT-QA-0051 Rev. 7.0, 10/02/06)

Prepared by: _____

Reviewed by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental, Health and Safety Coordinator

Approved by: _____
Laboratory Director

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1. PURPOSE AND APPLICATION

1.1. This SOP details procedures for receiving samples into the laboratory and describes laboratory custody practices.

1.2. This protocol described here complies with accepted laboratory custody procedures and regulatory requirements.

2. RESPONSIBILITIES

2.1. It is the responsibility of the Sample Receiving Department to receive samples in full compliance with this SOP. Sample receiving duties of the sample custodian (and other designated individuals) are described in the "PROCEDURE" section. Following this SOP assures that all samples are properly received and will always be under the custody of a person or stored in a secure area while at the laboratory.

For the purpose of this SOP, a sample is 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 you locked it up, or

It is in a designated secure area. (Secure areas shall be accessible only to authorized personnel.)

2.2. It is the responsibility of each analyst or any TestAmerica Pittsburgh staff member to maintain sample custody for all entrusted samples as described herein in full compliance with this SOP.

2.3. It is the responsibility of the laboratory director, QA manager, and supervisors to assure that operations are conducted in full compliance with this SOP. It is also their responsibility to supply training, materials, and equipment so that the laboratory staff can comply with all SOP requirements.

3. SAFETY

3.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, the Waste Management SOP, and this document.

3.2. Sample bottles containing samples suspected to contain high levels of cyanide or sulfide shall be marked with a sticker marked "High Cyanide" or "High Sulfide". These sample bottles shall only be opened in a fume hood.

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3.3. 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.

3.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be

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.
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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

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worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately.

3.5. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred, and prepared in a fume hood or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made. If waste is generated during operations, it will be segregated and disposed of according to the facility hazardous wastes procedures as appropriate. The Environmental Health and Safety Coordinator will be contacted if related information is required.

3.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor or the EH&S coordinator.

4. PROCEDURE

4.1. Samples will be received at the laboratory facility during normal working hours (8 a.m. to 5 p.m.). If sample receipt is anticipated outside normal working hours, the project manager must notify and coordinate sample receipt with the sample custodian, who in turn will designate personnel to receive the samples. The sample custodian examines the shipping container (cooler, box, or other shipping container) and completes the header information on a Cooler Receipt Form (see “APPENDICES,” Section 8.0). Information recorded at this time will include: client name and project, quote number, TestAmerica lot number, the date the shipping container was received and temperature checked, and the date that the shipping container was unpacked.

The temperature of the cooler may be taken either by using an enclosed temperature blank, if supplied, or by inserting a thermometer between samples in the cooler (the thermometer must not come in contact with the ice). If the temperature of the cooler is not 4°C ±2°C, this must be noted on a Condition Upon Receipt Variance Report (see “APPENDICES,” Section 8.0). The temperature will be recorded on the Cooler Receipt Form. The reading that is recorded will take into consideration the relevant thermometer correction factor.

If the cooler temperature is >6°C, the sample custodian will document which sample containers were inside each specific cooler where the temperature was >6°C. This will be documented on the Condition Upon Receipt Variance Report and the project manager will be notified.

Other information to be noted at this time is: presence/absence of custody seal(s) on the shipping container(s), and their condition.

4.2. The sample custodian opens the shipping container and removes the entire contents, assuring that all enclosed samples and documents are retained. After reviewing the entire contents of the shipping container and carefully checking sample integrity (intact, broken,

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leaking, etc.), labels, and shipment completeness, the remaining information is placed on the Cooler Receipt Form (see “APPENDICES,” Section 8.0).

4.3. The sample custodian must compare the field chain(s) of custody, the sample labels, and airbills or bills of lading. If there are any inconsistencies between the forms, the discrepancies are documented on the Condition Upon Receipt Variance Report (see “APPENDICES,” Section 8.0). If samples are received out of holding time, a Nonconformance Memo must be completed and the project manager notified. The project manager then notifies the client and a decision is made concerning whether to proceed with analysis.

4.4. The sample custodian measures the pH of preserved aqueous samples (with the exception of samples that have been submitted for the testing of volatile organic compounds). This is done by placing a few drops of the sample on *narrow*-range pH paper using *either a disposable cup (for DOD clients) or* a clean disposable Pasteur pipette. This measurement determines whether the sample has been properly preserved. (For Oil & Grease pour in cap and then pipette.) The used pipette is then discarded. **Note:** The pH of samples to be analyzed for VOAs will be measured by the volatiles analytical group after removing an aliquot for analysis.

4.5. For all potentially chlorinated samples the sample custodian checks for residual chlorine by testing the sample with KI-starch paper. If residual chlorine is present, the sample custodian must indicate so on the sample condition upon receipt form and issue a NCM. Also, the client, the PM, and the group responsible for analysis of the sample must be notified. Steps for removal of residual chlorine are included in the SOP for the method the sample is to be analyzed by.

4.6. If all samples recorded on the field chain(s) of custody were received by the laboratory and there are no problems observed with the sample shipment, the sample custodian signs the field chain(s) of custody in the "Received for Laboratory by:" box on the document. Problems such as broken bottles and pH or temperature outside criteria are noted on the Condition Upon Receipt Variance Report (see “APPENDICES,” Section 8.0). Where approved by the project manager, unpreserved samples will be preserved by the sample custodian. This action will be documented on the Condition Upon Receipt Variance Report (CURV) and where required by the PM in a non-conformance memo. Where lab reagents are used to make a preservative adjustment, record the reagent, lot number and amount of reagent added in the comment field on the CURV report. Whenever the laboratory preserves samples for metals analysis, the samples must be held for 16 hours after preservation before analysis may be performed.

4.7. The project manager’s Quote form (the LIMS description of the sampling program) is reviewed to assure that the field chain(s) of custody is in agreement. If the documents do not agree, the project manager is notified of the discrepancy. Samples are not logged in until all discrepancies are resolved.

4.8. If it has been necessary to complete a Condition Upon Receipt Variance Report, this will be forwarded to the project manager for review. The project manager will determine the action required and contact the client as necessary or otherwise agreed.

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4.9. Once the project manager has resolved any discrepancies between the field chain(s) of custody and Quote and has taken the appropriate action for any Condition Upon Receipt Variance Report that has been generated, the samples are logged in using the Laboratory Information Management System (LIMS). A unique laboratory identification number is assigned to each sample (i.e., C6B060001-001: C denotes Pittsburgh; 6 references the year [1996]; B signifies the month [February]; 06 means the sixth day of the month; 0001 is the first lot logged in; and -001 is the first sample of that lot).

4.10. The Sample Control person will enter each sample into the laboratory computer (QuantIMS), where a unique lot number is assigned to each project received, and sequential sample numbers are designated for each client identification within the lot.

Lot Numbers: The lot number is nine characters in length and is based on the date of receipt. Lot number A5J010021 is described as follows:

A - TestAmerica location where the samples were received.
(A = North Canton, B = Tampa, C = Pittsburgh, etc.)
5 - Last digit of the year (i.e. 1995).
J - Month (i.e. A = January, B = February, J = October, etc.)
01 - The next 2 numeric characters identify the day of the month, in this case, the first day of the month.
0021 - The next 4 numeric characters are the sequential assignment of numbers specific to each lot received. Each day the first lot logged in receives the number "0001", the second lot receives the number "0002", etc..
For example:
If four bottles were submitted under Client ID numbers AB100-AB103 and the laboratory identification number generated by the computer is A2K100001, then the assigned laboratory number recorded on the Sample Log-In Sheet would be as follows:

Client ID Sample Number	Assigned Laboratory Number
AB 100	A2K100001-001
AB 101	A2K100001-002
AB 102	A2K100001-003
AB 103	A2K100001-004

Sample Numbers: The samples in each lot are assigned a sample number that is attached to the lot number and are reset at each new lot. For example: the first and second samples in the lot above are labeled A5J010121-001 and A5J010121-002.

Sample Suffixes: Each sample also has a 1 character field (which is not a required field for all samples) called the suffix, which identifies the sample as specified below.

Client Sample	No. Suffix
Method Blank	B
Laboratory Control Sample	C
Laboratory Control Sample Duplicate	L

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Matrix Spike	S
Matrix Spike Duplicate	D
Sample Duplicate	X
Serial Dilution	P
Sample Confirmation	Y
Post Digestion Spike	Z
Re-analysis	I

Example: A5J010121-001X is a sample duplicate for sample A5J010121-001.

Work Order Numbers: Each test requested by the client for an individual sample receives an individual 8 digit work order number assigned by QuantIMS. Work order number A5WE1-2-1C is described as follows:

A5WE1 - In addition to the three digit sample (i.e. - 001 and - 002), the first 5 characters of the work order number also identifies each unique sample. This identification is generated in QuantIMS using a sequential logic.

2 - The "modifier" indicates the type of run. In this case this is the second time the sample had to be run. If it needs reprepared and run again, the number would indicate a "3". The original analysis work order number assigns "1" to the modifier position.

1C - The "suffix" is the identification of the specific test for that sample. The suffix in this case is not always sequential, but is unique to the test to be performed on the sample.

4.11. Worksheets and sample container labels are printed. The sample custodian will assure that each sample container is labeled with a unique laboratory identification number that cannot be accidentally removed in the laboratory.

The label will contain the following information:

The laboratory information management system unique Lab ID number.

Sampling date.

Client ID.

Storage location.

Client code.

Number and size of bottles received.

4.12. The sample custodian will put the samples into secure storage areas. Samples for tests other than volatile organics are placed on numbered shelves inside secure walk-in refrigerators. Samples received for volatile organics are placed in separate secure

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refrigerators that have been designated for volatile sample storage only and are located within the volatiles lab area. From the time of login and refrigerator assignment by the sample receiving personnel, the volatiles personnel become the designated sample custodians for the volatile samples. As custodians they must document custody within their workgroup. In both cases temperature is maintained at 4°C ±2°C. The storage location for all received samples is entered in the laboratory information management system.

4.13. The sample custodian files the shipping documents, field chain(s) of custody, quote, related correspondence, cooler receipt form, analytical summary, along with preliminary invoice. The summary and supporting documentation are reviewed for accuracy by the sample custodian and placed in a project folder before being submitted to Project Management. Laboratory information management system worksheets are generated by the sample custodian and distributed to the analytical group leaders.

4.14. Internal Chain of Custody

The sample custodian transfers the custody of requested raw samples to the analytical staff using a Sample Custodian Removal Request (see “APPENDICES,” Section 8.0), which lists the laboratory identification number of the samples being transferred. The sample custodian checks that the sample labels and Sample Custodian Removal Request are in agreement, dates, records the time of transfer (using military time), and signs the Sample Custodian Removal Request as having relinquished the samples. The analyst will then sign the Sample Custodian Removal Request as having received the listed samples.

The Sample Custodian Removal Request form must accompany the raw samples while they are being analyzed. The analyst may transfer the samples to a second analyst using a second Sample Custodian Removal Request form that has been generated listing the samples and purpose of the transfer. All samples listed on the initial Sample Custodian Removal Request must be present and listed on the second Sample Custodian Removal Request. Transferring less than all of the samples listed on the initial Sample Custodian Removal Request is prohibited. If only several of the samples listed on the initial form are required, then all samples on the initial form will be returned to the laboratory sample custodian who will relinquish only those samples required, as described in the above section. The relinquishing analyst assures that all samples are present and correctly identified on the second Sample Custodian Removal Request before signing, dating, and recording the time of transfer (military time) on the second Sample Custodian Removal Request form. The second analyst signs the second Sample Custodian Removal Request form to accept the samples. The original Sample Custodian Removal Request from the laboratory sample custodian must be relinquished to the second analyst and will remain with the samples.

Custody of raw samples may be maintained if the analyst receiving custody as described above places the samples in a secure storage area accessible only to authorized personnel for the purpose of analysis. Storage conditions must be such that the integrity of the sample will be maintained for the required analyses.

Raw samples (whether the containers have sample volume remaining or are empty) shall be returned to the sample custodian with the original Sample Custodian Removal Request and any additional Sample Custodian Removal Requests that were required to document

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subsequent transfers. The Sample Custodian Removal Request must be reconciled with the samples being returned before the sample custodian accepts receipt of the samples. (The sample custodian will not accept the samples unless all of the containers listed on the Sample Custodian Removal Request are present.) The analyst then signs as relinquishing the samples, and sample custodian accepts custody of returned raw samples by signing, dating, and recording the time of receipt (using military time) on the Sample Custodian Removal Request that documents the most recent transfer of the samples.

The sample custodian will record which samples have been completely expended in analyses by making an entry on the Sample Custodian Removal Request.

Custody information for extracts, distillates, and digestates prepared from raw samples shall be recorded on the sample preparation benchsheet used to document the preparation. (A Sample Custodian Removal Request will not be used for sample preparations.) The individual who has conducted the preparation is responsible for completing custody transfer information. The relinquishing analyst assures that all sample preparations are present and correctly identified before signing, dating, and recording the time of transfer (military time) on the benchsheet. Prepared samples are transferred to and kept in a secure area accessible only to authorized personnel for the purpose of analysis. If further custody transfers are necessary, these are recorded in a similar manner on the same benchsheet. The benchsheet with completed custody information must accompany the prepared samples whenever custody transfers are necessary.

4.15. Samples will be properly disposed of by the sample custodian thirty days after the final report unless the laboratory has been otherwise notified in writing. All samples in a single TestAmerica laboratory lot will be disposed of at the same time, and a record documenting the lot disposal will be kept in a logbook maintained by the sample custodian.

4.16. The temperature of each walk-in cooler is monitored daily Monday through Saturday using a calibrated mercury bulb thermometer.

4.17. **Cooler Handling Procedure**: All coolers should be rinsed out after receipt and unloading. If a cooler smells or is visibly soiled it should be soaped and warm water rinsed. If a cooler cannot be cleaned up or remains odorous after cleaning, it should be discarded.

4.18. Any deviation from the conditions and handling described in the “PROCEDURES” section will be documented using a Nonconformance Memo (NCM) with distribution to the Project Manager and Quality Assurance Department. Corrective action must be fully explained in the Nonconformance.

5. DEFINITIONS

5.1. Custody: The definition of custody may be found in Section 2.1.1.

5.2. Internal Chain of Custody: Records generated by various departments internal to the laboratory and which document custody of samples or sample preparations for transfers within the laboratory.

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5.3. LIMS: Laboratory Information Management System. The computer system used to track information about samples after being received by the laboratory.

6. POLLUTION PREVENTION

6.1. 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."

7. WASTE MANAGEMENT

7.1. There are no special waste streams associated with this procedure.

8. APPENDICES

8.1. Disaster Recovery Plan Appendix

8.2. Cooler Receipt Form, Page 1 of 2 (example form). Cooler Receipt Form, Page 2 of 2 (example form).

8.3. Sample Acceptance Policy

8.4. Condition Upon Receipt Variance Report (example form).

8.5. Sample Custodian Removal Request (example form).

8.6. Holding Times are noted in the TestAmerica Pittsburgh Laboratory Quality Manual (LQM), current version. Please note, client specific or project specific holding times would supersede the holding times listed in the LQM.

9. REFERENCES

9.1. TestAmerica Pittsburgh LQM, current version.

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Disaster Recovery Plan Appendix

1. When sample location transfers are required due to cooler malfunction or power failure, all transfers must be properly documented as described in the above SOP sections. Sample security must be properly maintained with access to samples limited, controlled, and documented. Temperature of the relocated samples will be documented and controlled to 4°C ±2°C.

2. In the event of a power failure limiting available cold storage capacity to less than that required to adequately store all samples at the necessary temperature, backup refrigeration trucks will be called to give assistance on site to store samples and maintain proper temperature at 4°C ±2°C. Trucking companies that could be called are:

   Thermo King Transport Refrigeration
   Ryder Trucks
   Budget Trucks

3. If refrigeration trucks are not available during a power failure, dry ice will be used to maintain proper temperature.

4. When cooling units malfunction, a local heating, ventilation, and air conditioning contractor will immediately be called to correct the problem. Sample receiving will coordinate all service calls using a 24-hour servicing company to minimize impact on sample integrity.

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TestAmerica Pittsburgh
Sample Acceptance Policy

NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. TestAmerica PITTSBURGH will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.

When completing the chain of custody form, please do not forget to sign your name in the "relinquished by" box.

NELAC requirements are as follows:

- Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided.
- Each sample shall be labeled with unique, durable and indelible identification,
- The samples shall be collected in the appropriate sample containers.
- The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
- Sufficient sample volume must be available to perform the requested analyses.
- If *Matrix Spikes* are required for your project, separate sample volumes must be available for the requested analyses
- The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation.
- Samples must be preserved as specified in the requested methods
- Efforts should be made to minimize any air bubbles in aqueous volatile samples. Air bubbles also the escape of volatile organics. This is especially important because air bubbles tend to form in iced samples. Volatile vials containing air bubbles larger than a pea will be treated as non-conformances.
- Samples that required chilling must be received at < 6 ° C or they will be narrated as non-conforming samples.

NOTE: Never affix a label directly on an Encore Volatile sampler.

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Cooler Receipt Form
STL Pittsburgh

Client: _____ Project: _____ Quote: _____

Cooler Rec'd & Opened for Temp. Check on: _____

Coolers Opened and Unpacked on: _____ By: _____
(Signature)

STL Pittsburgh Lot Number: _____

		Yes	No
1.	Were custody seals on the outside of the cooler?		
	If YES, how many and where? Quantity __ Location		
	Were signatures and date correct?		
2.	Were custody papers included inside the cooler?		
3.	Were custody papers properly filled out (ink, signed, match labels)?		
4.	Did you sign the custody papers in the appropriate place?		
5.	Was shippers packing slip attached to this form?		
6.	Were packing materials used?		
	If YES, what type?		
7.	Were the samples chilled? (Record temperatures on reverse side.)		
8.	Were the samples appropriately preserved?		
9.	Were all bottles sealed in separate plastic bags?		
10.	Did all bottles arrive in good condition (unbroken)?		
11.	Were all bottle labels complete (sample ID, preservatives, etc.)?		
12.	Did all bottle labels and/or tags agree with custody papers?		
13.	Were correct bottles used for tests indicated?		
14.	Were all VOA vials checked for the presence of air bubbles?		
15.	Was a sufficient amount of sample sent in each bottle?		

16. Samples received by: FEDEX UPS CLIENT DROP-OFF OTHER DHL

Explain any discrepancies: _____

Level 2 Review _____

Was contacted on _____ by _____ to resolve discrepancies.

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Cooler Receipt Form

STL Pittsburgh

P: Preserved
UP: Unpreserved

Sample ID	TMET PH<2	DMET PH<2	HG PH<2	NUT(1) PH<2	CN PH ≥12	OG TPHC PH<2	PHEN PH<2	SULF PH ≥12	TOC PH<2	TOX PH<2	VOA P/UP	hrdnss PH<2	Cl_2 RES		

(1) "NUT" could include sample bottles for ammonia, chemical oxygen demand, nitrate/nitrite, TKN, or total phosphorus

Comments: _____

_____

_____

Cooler Number	Temperature*	Thermometer

*Acceptable Temperature Range: 4°C ± 2°C

Sample	Lot Number**

**Please use an asterisk if bottle lot number was covered by the label

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Condition Upon Receipt Variance Report
STL Pittsburgh Laboratory

Client: _____ Date: _____
Project No.: _____ Initiated by: _____
Analysis Requested: _____ RFA/COC: _____
Client Sample Numbers Affected: _____

Condition/Variance (Check all that apply):

1. ☐ Not enough sample received for proper analysis. Received approx. _____
2. ☐ Sample received broken/leaking.
3. ☐ Sample received without proper preservative.
   - ☐ Cooler temperature not within 4°C ± 2°C.
     - Record temperature: _____
     - ☐ pH _____
     - ☐ other: _____
4. ☐ Sample received in improper container.
5. ☐ Sample received without proper paperwork.

   _____
6. ☐ Paperwork received without sample.
7. ☐ No sample ID on sample container.
8. ☐ Custody tape disturbed/broken/missing.
9. ☐ Sample splits performed by lab.
10. ☐ Volatile sample received with approximately _____ mm headspace.
11. ☐ Sample ID on container does not match on paperwork. Explain:

    _____

    _____
12. ☐ All coolers on airbill not received with
13. ☐ Other (explain below):

    _____

    _____

    _____

Notes: _____
_____
_____

Corrective Action:

☐ Client's Name: _____ Informed verbally on: _____ By: _____

☐ Client's Name: _____ Informed in writing on: _____ By: _____

☐ Sample(s) processed "as is" _____

☐ Sample(s) on hold until: _____ If released: _____

Sample Control Supervisor Review: _____ Date: _____

Project Management Review: _____ Date: _____

SIGNED ORIGINAL MUST BE RETAINED IN THE PROJECT FILE

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SEVERN TRENT STL

STL PITTSBURGH
301 Alpha Drive
Pittsburgh, PA 15238
Phone: 412-963-7058
Fax: 412-963-2468

COC/Sample Request

PROJECT NAME **SITE**

Lot # / Sample#	Analysis	Matrix

Raw Sample	Raw Relinquished by				Raw Received by			
	Date	Time	Analyst	Location	Date	Time	Analyst	Location

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STL

Implementation Date: 5/28/07

STL PITTSBURGH STANDARD OPERATING PROCEDURE

TITLE: Bottle and Cooler Preparation

(SUPERSEDES: PITT-QA-0054, Revision 1)

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Laboratory Director

Proprietary Information Statement:

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1.0 PURPOSE AND APPLICATION

The purpose of this SOP is to describe the procedures used by STL-Pittsburgh to prepare and ship sampling kits for field use. A sampling kit contains the coolers, pre-cleaned containers, preservatives, and custody records necessary to complete a sampling event and return the samples to STL under conditions necessary to preserve the integrity of the samples.

2.0 RESPONSIBILITIES

2.1 Sample Receiving Area Leader: The sample receiving area leader is responsible to ensure that all sample receiving associates involved in shipping sample bottles and coolers containing the preservatives described in this procedure have been trained per the requirements of this procedure The sample receiving area leader must receive training on the requirements for shipping hazardous materials as required at 49CFR172.704 and, if shipping air per IATA, training per the IATA Dangerous Goods Regulations, Section 1.5. This training must be completed within ninety days of hire or transferred to this position. Until trained, the individual must directly supervised by a second trained.

2.2 Sample Receiving Associate: The sample receiving personnel are responsible for maintaining an adequate inventory of pre-cleaned bottles, clean coolers, and chemical preservatives, assembling material for shipment and tracking coolers. Sample receiving associates are only permitted to ship under the requirements of Section 4.3, Small Quantity Exceptions, of this procedure, and must be trained to the requirements of this procedure and 49CFR173.4. The sample receiving associate is not permitted to ship any other hazardous materials. This training must be completed within ninety days of hire or transfer to this position. Until trained, the individual must be directly supervised by a second trained individual.

2.3 All training described per 2.1 and 2.2 above must be repeated every three years.

2.4 Project Manager: The project manager is responsible for initiating the bottle order that identifies sufficient number of appropriate sample containers for the client's use.

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3.0 SAFETY

3.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.2 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
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.
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	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.
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.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even 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.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

3.3 Vials or bottles containing preservatives must be labeled with the name of the preservative contained in the vial or bottle.

3.4 A Material Safety Data Sheet must be provided for each preservative contained in a vial or bottle when shipped to a client.

4.0 PROCEDURE

4.1 Requirements for Shipping DOT Hazardous Materials and/or IATA Dangerous Goods

4.1.1 Any material meeting the definition of one of the nine DOT hazard classes is considered a hazardous material when offered for domestic shipment by ground, rail, air, or vessel.

4.1.2 Any material meeting the definition of a one of the nine IATA hazard classes is considered a Dangerous Good when offered for transportation by air internationally. Note: A common carrier may require a domestic air shipment of a DOT hazardous material to be shipped in accordance with IATA regulations.

4.1.3 The following are the nine DOT and IATA hazard classes:

4.1.3.1 Explosives (Class 1)

4.1.3.2 Compressed gases (Class 2)

4.1.3.3 Flammable liquids (Class 3)

4.1.3.4 Flammable solids, spontaneously combustible, and dangerous when wet compounds (Class 4)

4.1.3.5 Oxidizers and peroxides (Class 5)

4.1.3.6 Poisons or toxins (Class 6)

4.1.3.7 Radioactive materials (Class 7)

4.1.3.8 Corrosive materials (Class 8)

4.1.3.9 Miscellaneous materials (Class 9)

4.1.4 There are seven preservatives that are routinely added to samples that are a DOT "Hazardous Material" and/or IATA dangerous goods when offered for transportation. They are Hexane, Nitric Acid, Sulfuric Acid, Hydrochloric Acid, Sodium Hydroxide, Methanol, and Sodium Bisulfate (not currently used at Pittsburgh). These materials may be shipped domestically using the provision of 49 CFR 173.4 or the IATA provisions for "Dangerous Goods in Excepted Quantities". International shipments must be shipped under the IATA provisions.

4.1.5 Any sample bottle or vial containing any of the materials listed in 4.1.4 above and being shipped or delivered to a client, service center or STL lab must be shipped in accordance with full DOT regulations unless shipped using one of the following two exceptions:

4.1.5.1 Materials of Trade per 49 CFR 173.6

4.1.5.2 Small Quantity Exceptions per 49 CFR 173.4

4.1.6 Preservatives, other than those listed in 4.1.4 above, shall not be used unless approved by the EHSC/EHSD.

4.2 Materials of Trade Exception

4.2.1 Under 49 CFR 173.6, the samples that are analyzed by the laboratory are classified as a "Material of Trade." Under the provisions of 49 CFR 173.6, "Materials of Trade" are not subject to the provisions of the hazardous materials shipping regulations as long as the following provisions are met.

4.2.1.1 The material is transported by STL's employees, the client's employees, or private courier hired by STL or the client.

4.2.1.2 The total gross aggregate weight of the sample package does not exceed the limits set forth in the citation. Individuals need to check the regulatory citation since the total mass varies by hazard class and packing group

4.2.1.3 The total gross aggregate weight of all packages containing known hazardous materials does not exceed 440 pounds.

4.2.1.4 The materials are packaged in accordance with the citation. Packaging for each classification of material may vary slightly. However, in general the packages must be leak tight for liquids and gases, sift proof for solids, be securely closed, secured against movement, and protected against damage.

4.2.1.5 The outer packages are marked with either a common name or a proper shipping name. Note: This citation does not apply to explosive (Class 1) or radioactive (Class 7) materials.

4.2.1.6 The operator of a motor vehicle that contains a material of trade must be informed of the presence of the hazardous material.

4.2.2 When using this exception, there are limitations on the quantity of hazardous materials permitted in individual containers for the various hazard classes. Contact the EHSC prior to using this exception.

4.3 Small Quantity Exception

4.3.1 Under 49 CFR 173.4, sample kits containing hazardous materials listed in 4.1.3 above are not subject to the provisions of the DOT hazardous materials shipping regulations or IATA dangerous goods regulations (Section 2.7) as long as the following provisions are met.

4.3.1.1 The amount of material in each inner package may not exceed 30 ml or 30 g.

4.3.1.2 The inner package must either be plastic having a minimum thickness of no less than (0.2) millimeters (0.008 in), or earthenware, glass or metal.

4.3.1.3 The inner package must be packed with a secure material that will not react with the material in the container and will absorb all liquid present.

4.3.1.4 The inner packages must be packed in a strong outer package that can withstand the drop and stack tests specified in the citation. The package must be able to be dropped from 5.9 feet on any corner or side without any containers breaking or leaking and must be able to withstand being stacked to a height of ten feet for 24 hours without collapsing.

4.3.1.5 The total gross aggregate weight of each package may not exceed 64 pounds.

4.3.1.6 International shipments must not exceed the limits for each outer package specified under the IATA table.

4.3.1.7 If hazardous materials are shipped under the provisions of 49 CFR 173.4, the following statement, “This package conforms to 49 CFR 173.4” must be included on the outside of the package (see 8.1.1 for sample marking).

4.3.1.8 If the sample kits are shipped under the provisions of the IATA regulations, a “Dangerous Goods in Excepted Quantities” label must be

completed and attached (see 8.1.2 for sample label). Sample kits shipped internationally must be shipped under the IATA provisions.

4.3.1.9 ***Nitric acid in concentrations greater than 20% is forbidden to be shipped via air using the 49 CFR 173.4 exception. Shipment of this material must be made by ground only.***

4.3.1.10 ***Nitric Acid is not permitted to be shipped via air using the "Dangerous Goods in Exempt Quantities" provisions.***

4.3.1.10.1 ***Nitric Acid in concentrations of 20% or less may be shipped by air under the IATA provisions. The maximum amount of material in the inner packages may not exceed 500ml.***

4.4 Requirements for Shipping Known Samples of Hazardous Waste

4.4.1 Under the provisions of 40 CFR 261.4(d) samples are excluded as "Hazardous Waste" as long as they meet the following requirements.

4.4.1.1 The sample is being transported to a laboratory for the purpose of testing.

4.4.1.2 The sample is being transported back to the sample collector after testing.

4.4.1.3 The sample is being stored by the sample collector prior to transportation to a laboratory for testing.

4.4.1.4 The sample is being stored in a laboratory before testing.

4.4.1.5 The sample is being stored in a laboratory after testing but before it is returned to the sample collector.

4.4.1.6 The sample is being stored temporarily in the laboratory after testing for a specific purpose (for example, until conclusion of a court case or enforcement action where further testing of the sample may be necessary).

4.4.2 As long as the sample does not meet one of the other definitions of a "Hazardous Material" under DOT regulations it is not a DOT "Hazardous Material".

4.5 Sample Bottles Filled by Clients or STL associates

4.5.1 As mentioned, the seven DOT "Hazardous Materials" that are used as preservatives are Hexane, Nitric Acid, Sulfuric Acid, Hydrochloric Acid, Sodium Hydroxide, Sodium Bisulfite and Methanol.

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4.5.2 With regard to the acids and bases that are used to preserve samples, as long as the amount of preservative in the original empty sample container does not exceed the amount listed below, the sample will not meet the definition of a DOT corrosive material when filled with water by the client or an STL associate.

4.5.3 Samples preserved with Methanol and Sodium Bisulfate and wipe samples preserved with Hexane are still DOT "Hazardous Materials". They may be shipped under the provisions for hazardous materials in excepted quantities as long as there is less than 30 ml per container being returned to the lab. The volume in the container does not include soil added to the vials with the Methanol. However, it does include the volume of water added to the vials containing Sodium Bisulfate.

4.5.4 The volume of preservative in a sample bottles shall not exceed those listed in the table below. Preservative volumes shall be adjusted proportionally for containers sizes not listed below.

Preservative	Sample Container Size	Maximum Volume
Sulfuric Acid (1:1 Concentrated acid in water)	1 Liter	4 ml
Nitric Acid (1:1 Concentrated acid in water)	1 Liter	6 ml
Nitric Acid (1:4 Concentrated acid in water)	1 Liter	15 ml
Hydrochloric Acid (1:1 Concentrated acid in water)	40 ml VOA vial	0.7 ml
Sodium Hydroxide (50% by weight in water/Approximately 10N)	500 ml	1.25 ml
Hexane	40ml VOA vial	30ml
Methanol	40ml VOA vial	30ml

4.5.5 Wipe samples preserved with 0.1% nitric acid will not meet the definition of a DOT hazardous material.

4.6 Shipping Sample Kits to Clients, Service Centers or Other STL Labs

4.6.1 Bottle Order Notification

4.6.1.1 The project manager or designee enters sample container requests into the LIMS. The request form is routed to the shipping department for preparation of the sampling kits.

4.6.1.2 Notify the project manager or assistant immediately if a sampling kit request cannot be completed.

4.6.2 Preparation of Sampling Kits

4.6.2.1 Pre-cleaned Bottles, and Preservatives

4.6.2.1.1 All sample containers provided by STL Pittsburgh are new.

4.6.2.1.2 Preservatives may either be purchased in prepackaged vials or bottles or added to vials or bottles by a trained STL associate. The vendor specifications meet analytical requirements for cleanliness. If preservatives are purchased prepackaged, the volumes and concentrations shall not exceed those listed in the table in 4.6.4.2 below.

4.6.2.1.3 Documentation certifying sample cleanliness must be maintained by the laboratory or the vendor, and can be provided to the client upon request.

4.6.3 Cleaning Coolers

4.6.3.1 Remove old tape and old labels.

4.6.3.2 Wipe down the inside of the cooler with wet paper towels. Remove any visible dirt or foreign material by scrubbing with hot water. In cases where the cooler has been heavily contaminated (e.g., oil or solvent has been spilled in the cooler), the cooler should be replaced.

4.6.3.3 Dry the coolers.

4.6.3.4 If an STL cooler does not have STL's name and address printed on the cooler, that information is printed on the cooler.

4.6.4 Filling the Bottle Order

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4.6.4.1 The sample receiving associate fills the bottle order with the requested bottles and preservatives.

4.6.4.2 When filling sample bottles or vials with preservative, the maximum volume of preservative in each container shall not exceed the values listed in the following table:

Preservative	Sample Container Size	Maximum Volume	Hazard Class	ID#	Mode
Sulfuric Acid (1:1 Concentrated acid in water)	250 ml	1 ml	8	UN1830	Air or Ground
Nitric Acid (1:1 Concentrated acid in water)	500 ml 250 ml	3 ml 1.5 ml	8	UN2031	Ground Only
Nitric Acid (1:4 concentrated acid in water)	500 ml 250 ml	7.5 ml 3.75 ml	8	UN2031	Cargo Air or Ground
Hexane	40 ml VOA vial	30 ml	3	UN1208	Air or Ground
Hydrochloric Acid (1:1 Concentrated acid in water)	40 ml VOA vial 1 Liter	0.7 ml 17.5 ml.	8	UN1790	Air or Ground
Methanol	40 ml VOA vial	30 ml	3	UN1230	Air or Ground
Sodium Hydroxide, 50% by weight, approximately 10N	500 ml 250 ml	1.25 ml 0.625 ml	8	UN1824	Air or Ground
Sodium Hydroxide, 4 N	500 ml 250 ml	3 ml 2 ml	8	UN1824	Air or Ground

4.6.4.3 Parts of the above table were derived from the table listed in 4.5.2 above. The sample container sizes and maximum volumes are based on the sample container sizes used at STL-Pittsburgh.

4.6.4.4 A trip blank must accompany each cooler as indicated by the client. The VOA trip blanks are labeled with the words “trip blank.” The water used for trip blanks is taken from the GCMS volatiles sample preparation area. It is the same water source that is used for GCMS volatiles analysis methods blanks and holding blanks.

4.6.4.5 A temperature blank is provided with each cooler requiring sample temperature preservation. The temperature blanks are labeled “temp blank.”

4.6.4.6 Containers are packed in boxes or coolers.

4.6.4.6.1 Boxes may be used only for nonpreserved containers. All preserved containers will be packaged in coolers.

4.6.4.6.2 Cardboard dividers, bubble wrap, and foam sheets or sleeves shall be used to prevent breakage.

4.6.4.6.3 Packing will be done so that no glass container is touching another container or the cooler side. Foam sheets, bubble wrap, or 3M absorbent sheets (or equivalent) will be used to prevent breakage during shipment.

4.6.4.6.4 Wrap all glass containers in bubble wrap or bubble baggies prior to packing. Plastic containers may be placed in the coolers without bubble wrap but still must be securely positioned to minimize movement during shipment.

4.6.4.6.5 When preservative vials and bottles are being shipped the following additional steps must be taken:

4.6.4.6.5.1 A layer of absorbent sheet is placed into the cooler.

4.6.4.6.5.2 A plastic bag is placed inside the cooler.

4.6.4.6.5.3 The containers and packing material are then placed inside the bag.

4.6.4.6.5.4 Additional bubble wrap is added to the cooler to fill all gaps and prevent movement of the vials and bottles during shipment.

4.6.4.6.5.5 The outside of the cooler must be marked with the words "This package conforms to 49 CFR 173.4" or if shipped in accordance with the provisions of the IATA regulations, a "Dangerous Goods In Excepted Quantities" label must be completed and attached to the cooler. When completing the IATA label, refer to the table in 4.6.4.2 above for the hazard class(es) and ID number(s) to be entered on the label. Sample kits shipped internationally must be shipped under the IATA provisions.

4.6.4.7 Place the following paperwork in the cooler or box:

4.6.4.7.1 Sample labels for each bottle sent

4.6.4.7.2 Chain of custody forms, as required.

4.6.4.7.3 Cooler custody seals to be used by the client for the return shipment as required.

4.6.4.7.4 Copy of the bottle order.

4.6.4.7.5 Copy of material safety data sheet for each preservative used.

4.6.4.8 Close and secure the box or cooler with shipping tape.

4.6.4.9 Mark the outside of the cooler or box with the statement, "FOR CHEMICAL EMERGENCY CALL INFOTRAC / 1-800-535-0535, 24 HRS. A DAY / 7 DAYS A WEEK" (See 8.1.4 for example).

4.7 Preparing The Shipping Bill

4.7.1 Prepare the shipping bill using the appropriate software program (e.g., Federal Express' Café System). If the shipping bill is to be prepared manually, prepare the shipping bill according to the client's specifications, as follows:

4.7.1.1 An air bill must be completed with the sample receiving associate's name, date, telephone number, recipient's name, recipient's address, and the recipient's telephone number.

4.7.1.2 Confirm whether the charges are going to be billed to the sender or billed to the recipient's account number and check the appropriate box.

4.7.1.3 Check the appropriate box for the service requested (e.g., priority, standard, ground, or Saturday delivery).

4.7.1.4 Note if shipment contains dangerous goods or not.

4.7.1.5 Record the total weight and declared value, if any.

4.7.1.6 Keep one copy of the air bill and attach the rest of the air bill to the cooler as required by the freight company.

4.7.2 A copy of the bottle order form is filed in the sample receiving area.

4.8 Any unauthorized deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

5.0 DEFINITIONS

5.1 Chain-of-Custody form: A legal document demonstrating the physical custody and traceability of samples.

5.2 Cooler-seal: Seals used to demonstrate that custody has been maintained and that unauthorized personnel have not had access to the samples.

5.3 Trip Blank: A blank that accompanies the bottles and samples and is tested to ensure that the samples were not contaminated during shipment.

6.0 POLLUTION PREVENTION

6.1 Not Applicable

7.0 WASTE MANGEMENT

7.1 Not Applicable.

8.0 APPENDICES

8.1 Samples Markings and Labels

8.1.1 Sample 49 CFR173.4 Marking

8.1.2 Sample Dangerous Goods In Excepted Quantities Marking

8.1.4 Sample Emergency Notification Statement

FOR CHEMICAL EMERGENCY

CALL INFOTRAC / 1-800-535-5053

24 HRS A DAY / 7 DAYS A WEEK

9.0 REFERENCES

9.1 STL Pittsburgh Laboratory Quality Manual, current revision.

9.2 Memorandum from N. Nunn to EHSCs, GM's, LD, K. Wheatstone, C. Carter dated 4/17/02 concerning Shipment of Samples.

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STL

Implementation Date: 2/2/07

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

Title: Semi-Automated, Pyridine-Barbituric Acid for Total Cyanide (Method 335.2,335.4 & 9012A)

(SUPERSEDES: PITT-WC-0018, Rev. 9)

Prepared by: Erin M Bozik

Reviewed by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental, Health and Safety Coordinator

Approved by: _____
Laboratory Director

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1. SCOPE AND APPLICATION

1.1 This method is applicable to the determination of Total Cyanide in waters, solids, tissues and wipes. It is based on EPA Method 335.2, 335.4 and SW846 Method 9012A.

1.2 The reporting limit is 0.01 mg/L for waters, 0.50 mg/kg for solids and tissues and 1.0 ug/wipe for wipes.

1.3 For DoD QSM Version 3 requirements, refer to SOP PITT-QA-DoD-0001.

2. SUMMARY OF METHOD

2.1 For Total Cyanide: Cyanide, as HCN, is released from cyanide complexes by distilling/refluxing the sample with strong acid and absorbed in a sodium hydroxide solution.

2.2 The sodium hydroxide solution is analyzed colorimetrically on an autoanalyzer using the pyridine-barbituric acid method. The color is read at 575 nm.

3. DEFINITIONS

3.1 NELAC CCV: The NELAC CCV is a spiked laboratory control sample used to monitor the accuracy of the calibration curve. The NELAC CCV is analyzed after the calibration curve. The NELAC CCV must be prepared from a source independent of the calibration standards material. The NELAC CCV is not distilled.

3.2 LCS: The Laboratory Control Sample (LCS) is a spiked laboratory control sample used to monitor the accuracy of the analytical process independent of possible interference effects due to sample matrix. The LCS is processed through all method steps with the samples. The LCS must be prepared from a source independent of the calibration standard materials. Successful analyte recovery for the LCS provides assurance that the method is in control.

3.3 LCSD: The Laboratory Control Sample Duplicate (LCSD) is processed with the LCS when insufficient sample is not available to process a MS/MSD. The LCS/LCSD is used to demonstrate batch precision. An LCSD is only required for batch QC, when a MS/MSD is not present. The LCSD is prepared from the same materials as the LCS.

3.4 MB: A Method Blank (MB) is prepared with reagent grade water and all reagents used to process the samples. The MB is processed along with the samples through all procedures including distillation. It is used to monitor the laboratory and/or reagent contamination.

3.5 CCV: A Continuing Calibration Verification (CCV) Standard assures that the method calibration is in control. A CCV is run at intervals of up to ten samples within a sequence. The CCV may be prepared from the same source material as the calibration standard.

3.6 Continuing Calibration Blank (CCB): A 0.25 N NaOH solution. CCBs will be run immediately following each NELAC CCV and CCV. An instrument blank must be free of analyte down to the reporting limit. All reportable data must be bracketed by acceptable CCB samples.

3.7 MS: Matrix Spike (MS) is an aliquot of one sample in the QC batch that is spiked with a known amount of the target analyte. As a part of the QC batch, it accompanies the sample through all the steps of the analytical process.

3.8 MSD: Matrix Spike Duplicate (MSD) consists of a replicate portion of the sample which was designated as the MS. This portion is spiked and processed exactly as the MS.

3.9 MS/MSD results are used to determine the effects of the sample matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, the MS and the MSD results may not have immediate bearing on any sample except the one spiked.

3.10 QC Batch: QC batch is a set of 20 or fewer environmental samples plus associated laboratory QC samples that are similar in composition and that are processed within the same time period with the same reagents and standard lots. Laboratory QC samples such as LCS, matrix QC samples, and blanks are not included in the sample count for batching purposes.

3.11 Reagent Grade Water: Laboratory water which is produced by a Millipore DI system or equivalent. Reagent grade water must be free of the analyte of interest as demonstrated through the analysis of MBs.

4. INTERFERENCES

4.1 Nitrite and nitrate interfere, but are eliminated by the addition of sulfamic acid.

4.2 Chlorine interference can be removed by dropwise addition of sodium arsenite to the sample until there is no reaction to KI-starch paper. This is done upon notification from the sample custodian. The analyst will then document the removal of residual chlorine on the sample condition upon receipt form.

4.3 Sulfide interference can be removed by adding an excess of bismuth nitrate to the sample before distillation. This is done for Method 9012A. For Method 335.2 and 335.4, sulfides

are removed by the addition of cadmium or lead carbonate until there is no reaction to lead acetate paper.

5. SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, the facility addendum to the CSM, and this document.

5.2 **Potassium cyanide and sodium cyanide will give off Hyrdogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.**

5.3 Latex, vinyl, Nitrile or similar gloves may be used.

5.4 Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.5 If samples are identified with cyanide concentrations equal to or greater than 200 mg/L, immediately notify the department manager and personnel responsible for hazardous waste shipping. Those samples must be identified as extremely hazardous for other chemists and must receive special attention during disposal.

5.6 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
Acetic Acid (1)	Corrosive Poison Flammable	10 ppm-TWA	Contact with concentrated solution may cause serious damage to the skin and eyes. Inhalation of concentrated vapors may cause serious damage to the lining of the nose, throat, and lungs. Breathing difficulties may occur.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as potential health hazard; do not ingest.
Bismuth Nitrate	Oxidizer	None	May cause irritation to the respiratory tract, skin and eyes.
Chloramine-T	Irritant	None listed	May cause irritation to the mucous membranes and upper respiratory tract, skin and eyes.
Hydrochloric Acid (1)	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.
Lead Carbonate	Poison Neurotoxin Irritant Probable carcinogen Reproduc-tive hazard	0.05mg/m3 TWA as Lead	Inhalation can cause local irritation of bronchia and lungs and can cause symptoms such as metallic taste in the mouth, chest and abdominal pain. Skin contact can cause local irritation, redness and pain. Can be absorbed through the skin.
Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	**This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death.** Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heart beat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Potassium Hydroxide	Poison Corrosive	2 mg/m3 PEL Ceiling	Symptoms of inhalation may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes with tearing, redness, swelling.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Silver Nitrate	Poison Corrosive Oxidizer	0.01 mg/m3 TWA	Symptoms of inhalation may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. Symptoms of skin contact include redness, pain, and severe burns; eye contact causes blurred vision, redness, pain, severe tissue burns and eye damage.
Sodium Arsenite	Poison Reproduc-tive Hazard Carcinogen	None listed	Symptoms of inhalation include respiratory tract irritation. Causes skin irritation, and may be fatal if absorbed through skin.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid (1)	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.
Zinc Acetate	Irritant	None Listed	Symptoms of skin or eye contact include redness, itching and pain.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.7 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.8 VITON gloves may be worn when halogenated solvents are used for extraction or sample preparation, nitrile gloves may be used when other solvents are handled. Note: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.

5.9 Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made. Preparation of sodium hydroxide solutions produces considerable amounts of heat. Use plastic containers to mix this solution if possible. If glass containers are used, they must be free of any cracks or irregularities.

5.10 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit. Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates.

5.11 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1 Cyanide Distillation Apparatus – Midi "Easy-Dist System"

6.2 Vacuum Pump

6.3 Graduated Cylinders: Various sizes

6.4 Volumetric Flasks: Various Sizes

6.5 Automated Pipette

6.6 Balance: Accurate to 0.1 mg

6.7 Konelab Autoanalyzer: See manufacturer for requirements

6.8 Lead Acetate Indicator Paper

6.9 pH paper

6.10 Disposable 120 ml containers.

7. REAGENTS AND STANDARDS

7.1 Use reagent grade water (18 Mohm) for all solutions.

7.2 Sodium Hydroxide (0.25 N): This is purchased commercially, as an alternative it may be prepared as follows: In a 1L volumetric flask, dissolve 10.0g NaOH in approximately 900mL reagent-grade water. Dilute to volume with reagent grade water and mix thoroughly. Store in a plastic bottle.

7.3 Phosphate Buffer 1.0 M: This is purchased commercially, as an alternative it may be prepared as follows: In a 1L volumetric flask, dissolve 138g KH_2PO_4 in approximately 800mL reagent-grade water. Dilute to volume with reagent grade water and mix thoroughly.

7.4 Chloramine-T: In a 100 ml volumetric flask, dissolve 1.0g of chloramine-T hydrate in reagent-grade water. Dilute to volume with reagent grade water and mix thoroughly. **Prepare fresh weekly.**

7.5 Pyridine - Barbituric Acid Reagent: In the fume hood, place 15g barbituric acid in a 1L volumetric flask and add approximately 250 mL of reagent grade water, rinsing down the sides of the flask to wet the barbituric acid. Add 75 mL pyridine (C_5H_5N) with stirring and mix. Add 15 mL concentrated hydrochloric acid (12 M HCl) and mix until the barbituric acid is dissolved. Dilute to volume with reagent grade water and mix thoroughly.

7.6 Standards Preparation:

7.6.1 Stock Standard 1000 mg CN/L equivalent: This is purchased commercially, as an alternative it may be prepared as follows: In a 1L volumetric flask, dissolve 2.0g potassium hydroxide (KOH) in approximately 500 mL reagent grade water. Add 2.51g potassium cyanide (KCN) and mix until dissolved. CAUTION: KCN IS HIGHLY TOXIC. AVOID INHALATION OF DUST OR CONTACT WITH THE SOLID OR SOLUTIONS. Dilute to volume with reagent grade water and mix thoroughly. Standardize this solution weekly. Note: Prepare in a fume hood.

7.6.2 Cyanide Solution 1 (Curve,CCV) 10 ppm: This is prepared as follows: Pipette 1mL of the Stock Standard (7.6.1) into a 100mL volumetric flask containing 50 ml of 0.25 N NaOH. Dilute to volume with 0.25 N NaOH and mix thoroughly.

7.7 Working Standards: Set of six. The standard curve may not have fewer than five standards.

7.7.1 Standard Curve for Cyanide: 0.5, 0.25, 0.1, 0.05, 0.01 and 0.005 mg/L. Using a calibrated automatic pipette, pipette 5.0, 2.5, 1.0, 0.5 and 0.1 of the working stock standard (7.6.2) into 100 mL volumetric flasks. For the 0.005 mg/L standard pipette 5.0

ml of the 0.1 mg/L standard into a 100 ml volumetric flask. Bring to volume with .25 N NaOH and mix thoroughly. The standard solutions are 0.5, 0.25, 0.1, 0.05, 0.01 and 0.005 mg/L respectively. For this method the 0.25 N NaOH is used for the 0 mg/L standard.

7.7.2 To verify the efficiency of the distillation procedure, 0.05 and 0.25 mg/L standards will be distilled and checked against the curve. The recovery must be within ±10 percent.

7.7.3 Independent Stock Standard: 1000 mg CN/L. Prepare and standardize as in 7.6.1 using a different lot number.

7.7.3.1 Cyanide Solution 2 (LCS, MS & MSD) 10ppm: Prepare as in 7.6.2 using the 1000 ppm standard in 7.7.3.

7.7.3.2 NELAC CCV: 0.20 mg/L Pipette 2.0 ml of the 10 ppm (7.7.3.1) into a 100 ml volumetric flask. Bring to volume with 0.25 N NaOH and mix.

7.7.3.3 CCV (Continuing Calibration Verification): Add 1.0 ml of 7.6.2 10 ppm standard to a 100 ml volumetric flask. Bring up to volume with .25N NaOH and mix thoroughly. This is the 0.1 mg/L standard from the calibration curve.

7.8 Reagents for Distillation of Total Cyanide

7.8.1 Sodium Hydroxide 0.25N: See Section 7.2

7.8.2 Concentrated H_2SO_4

7.8.3 Magnesium Chloride Solution: 510g of $MgCl \bullet 6H_2O$ dissolved and diluted to 1 Liter with reagent grade water in a volumetric flask. As an alternative, a $MgCl \bullet 6H_2O$ solution can be purchased.

7.8.4 Bismuth Nitrate Solution: Dissolve 30.0g of $Bi(NO_3)_3 \bullet 5H_2O$ in 100 mL of reagent grade water. Continue to stir solution and add 250 mL of glacial acetic acid. Stir until dissolved. Dilute to 1 liter with reagent water. (Method 9012A).

7.8.5 Sulfamic Acid

7.8.6 Powdered cadmium or lead carbonate. (Method 335.2 and 335.4).

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Solid samples are unpreserved. Water samples are preserved with NaOH to a pH >12. All samples are stored at 4°C in plastic or amber glass containers.

8.2 The holding time is 14 days from date of sampling.

9. QUALITY CONTROL

9.1 The QC batch is defined in Policy #: QA-003: 'The QC batch is a set of up to 20 field samples plus associated laboratory QC samples that are similar in composition (matrix) and that are processed within the same time period using the same reagent and standard lots.' QC The QC batch includes a method blank and LCS/LCSD pair or method blank LCS/MS/MSD. It would also include and field QC such as field blanks, rinsate blanks, and/or field duplicates.

9.2 A Laboratory Control Sample is processed on each day of analysis with each batch of 20 or fewer environmental samples. The LCS is prepared from a standard source independent of the material used for instrument calibration. The LCS recovery for Total waters (Method 335.4) must be +/ – 10 percent of the true value. The LCS recovery for Total waters (Method 335.2 and 9012A) must be + /– 15 percent of the true value. The LCS for Total solids must be within the manufacturers control limits. If the LCS fails criteria, the analyst will check calculations and instrument performance and reanalyze the LCS once. If the LCS is still outside control limits, all samples in the QC batch will be reprepared and reanalyzed. If this is not possible due to limited sample quantity, the laboratory project manager will be notified and an analytical narrative provided with the data. If reprepared and reanalysis will be outside of holding time, the client should be notified and approval from the client must be obtained before reanalysis.

9.3 Method Blanks are processed and run at a frequency of one per QC batch. The method blank is run within the analytical sequence, that is bracketed by acceptable CCV's and CCB's. Method blanks must be free of analyte down to the reporting limit. **Refer to PITT-QA-DoD-0001 for specific DoD requirements for the method blank.** The method blank is analyzed along with the samples associated with it for reporting purposes. all reportable data must have an acceptable method blank. All samples within a QC batch, which is associated with an unacceptable blank, will be re-prepped and reanalyzed. If this is not possible due to limited sample quantity, the laboratory project manager will be notified and an analytical narrative provided with the data. If repreparation and reanalysis is outside of the holding time, the client should be notified and approval from the client must be obtained before reanalysis.

9.4 Continuing Calibration Verifications will be run after every ten or fewer samples. Acceptance limits are + or - 10 percent of the true value of the standard. The CCV may be prepared from the same material as the calibration curve. All reported data must be

bracketed by an acceptable calibration curve and a CCV, or by consecutive acceptable CCVs. Samples associated with an unacceptable CCV will be reanalyzed. If this is not possible due to limited sample quantity, the laboratory project manager will be notified and an analytical narrative provided with the data. If repreparation and reanalysis is outside of the holding time, the client should be notified and approval from the client must be obtained before reanalysis.

9.5 An MS/MSD pair is processed with every 10 environmental samples. The MS/MSD recoveries should be within + / - 25 percent of the true value, and the RPD between the MS and MSD should be + /- 20 percent. If the MS and the MSD fails recovery criteria, the analyst will check calculations and instrument performance, in order to evaluate evidence of matrix interference. If the LCS is acceptable and the MS/MSD are comparable and outside of QC limits, then a matrix effect is indicated and narrated accordingly. If the sample selected for spiking requires dilution because the initial concentration of the sample is excessively high, then the MS/MSD should be run at the same dilution as the unspiked sample. If the resulting spike is over range, this should be documented and narrated in the data report. The MS/MSD should never be run at a dilution greater than the associated sample as this alters the matrix of the sample and thereby makes it invalid.

9.6 A sample duplicate must be performed at a frequency of once per QC batch for DoD samples. The percent difference must be within ± 20%.

9.7 A Continuing Calibration Blank (CCB), which is 0.25 N NaOH will be run immediately following each NELAC CCV and CCV. An instrument blank must be free of analyte down to the reporting limit. All reportable data must be bracketed by acceptable CCB's. Samples associated with the unacceptable blanks will be reanalyzed. If this is not possible due to limited sample quantity, the laboratory project manager will be notified and an analytical narrative provided with the data. If repreparation and reanalysis is outside of the holding time, the client should be notified and approval from the client must be obtained before reanalysis.

9.8 Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any samples. MDLs are determined yearly. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in STL QA Policy: QA-005. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL reporting limit.

9.9 Initial Demonstration of Capability

Prior to analysis of any samples using this SOP, the following requirements must be met:

Initial Demonstration Study: This requires the analysis of four QC check samples. The QC check sample is a well-characterized, laboratory-generated sample used to monitor method performance, which should contain the analyte(s) of interest. The results of the initial demonstration study must be acceptable before analysis of samples under this SOP may begin. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP. Acceptance criteria for the LCS are given in Section 9.2.

10. CALIBRATION AND STANDARDIZATION

10.1 A standard curve is analyzed daily on the auto analyzer for cyanide. The correlation coefficient must be greater than or equal to 0.995.

10.2 The cyanide stock solution, 1000 ppm, must be initially standardized, then restandardized weekly or as needed based upon recovery of the LCS.

10.3 Cyanide stock standardization: Blank: Dissolve approximately 0.2 g KOH in 100 ml of water. Cyanide stocks: Add 5.0 mL CN stock 1000 mg/L. Dilute to 100 mL with reagent grade water. Add five drops of rhodanine indicator to all three beakers. Titrate with AgNO3 to a salmon-colored end point. Include brand name of chemical and lot number in the standard logbook. The 10 mg/L cyanide standards must be adjusted using the standardization stock values.

$$\frac{(A - B)\ 1000\ mg/L}{C} = \text{True Value of Stock}$$

A = Volume of $AgNO_3$ for titration of sample.
B = Volume of $AgNO_3$ for titration of blank.
C = CN - added to 100mL of reagent grade water = 5.0 mL.

10.4 The recovery of the distillation apparatus should be verified by preparing and distilling two standards equivalent to a high level (0.25 mg/L) and a low-level standard (0.05 mg/L) from the calibration curve. These distilled standards should be within 10% of the equivalent calibration standards. If not, corrective actions should be taken to improve recoveries before proceeding with sample preparation and analysis.

11. PROCEDURE

11.1 Total Cyanide:

11.1.1 Water LCS is prepared by taking 1.0 mL of the LCS/MS, MSD working stock (7.7.3.1) to 50mL reagent grade water. The theoretical value is 0.20 mg/L.

11.1.2 Soil LCS is purchased from independent vendor.

11.1.3 Matrix spike and matrix spike duplicate for waters: add 0.5mL of the working stock standard (7.7.3.1) to 50mL of sample. The theoretical value is 0.10 mg/L.

11.1.4 Soil matrix spike and matrix spike duplicate: Add 0.5mL of the working stock standard (7.7.3.1) to 1g of sample. The theoretical value is 5mg/kg.

11.1.5 For water samples (335.2, 335.4), pour 50mL of sample into a distillation flask. Check for the presence of sulfides with lead acetate paper. If sulfides are present, treat 25mL more of the stabilized sample (pH≥12) with powdered cadmium or lead carbonate until there is no reaction to the lead acetate paper. Filter the solution through a dry filter paper into a dry beaker. From the filtrate, measure the sample to be used for analysis. For water samples (9012A), add 5mL of the bismuth nitrate solution directly to the sample in the distillation flask if sulfide is detected using the lead acetate paper. Solid, tissue and wipe samples are not checked for sulfides until after the distillation is complete. If sulfides are detected in the distillate, it is treated with powdered cadmium or lead carbonate until there is no reaction to the lead acetate paper.

11.1.6 For solids and tissues, weigh 1.0g of sample and transfer to a distillation flask with approximately 50 ml of reagent grade water. For wipe samples, add the entire contents of the sample container to the distillation flask. For waters, transfer 50ml of sample to a distillation flask. Refer to PITT-QA-0024 for subsampling procedures. Add 50 mL of 0.25 N NaOH solution to the receiving flask. Assemble the cyanide distillation apparatus. Turn on the vacuum source and adjust the flow such that an even stream of air bubbles is in the scrubber tube. Through the inlet tube, add approximately 0.2g of sulfamic acid, and mix for three minutes prior to the addition of the 2.5mLs of concentrated sulfuric acid. Rinse the inlet tube with reagent water. Add 2.0mL magnesium chloride solution and rinse the inlet tube with reagent grade water.

11.1.7 Turn on the cooling water and the heat source. Heat the sample to boiling. The sample is distilled/refluxed under acidic conditions for 1.5 hours for Method 335.4 and 1 hour for Method 335.2 and 9012A. Be sure to adjust the airflow as necessary. After the heating period, turn off the heat and allow to cool for fifteen minutes. Keep the vacuum on.

11.1.8 Disconnect the scrubber and drain the sodium hydroxide solution into a 120mL disposable container. Be sure to properly label the container.

11.2 During analysis, the standards, LCS, blanks and samples are then combined with reagents. A color is produced by the specific analyte in the sample. The intensity of the color is

determined by the amount of analyte present. The color intensity is measured and converted to an electronic signal.

11.3 Consult the autoanalyzer operations manual for general operating procedures and recommended routine maintenance.

11.4 Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described

12. DATA ANALYSIS AND CALCULATIONS

12.1 Total Cyanide sample concentrations are computed by comparing sample peak heights with the standard curve using the following equation. Conversion of results to µg/L is accomplished, where necessary, by multiplication of results in mg/L by 1000.

For water samples: $C = A \times DF$

Where
C = Sample concentration in mg/L
A = Concentration from calibration curve in mg/L
DF = Dilution factor

For soil samples: $C = A \times DF \times S$

Where
C = Sample concentration in mg/kg, dry weight
A = Concentration from calibration curve in mg/kg
DF = Dilution factor
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation. The "S" factor is not used in the calculation of results for tissue samples.

For wipe samples: $C = A \times V \times DF \times 1000$

Where
C = Sample concentration in ug/wipe
A = Concentration from calibration curve in mg/L
V = Volume of distillate in L
DF = Dilution factor

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12.2 Percent Recovery (%R) Calculation:

$$\%R = \frac{\text{observed value}}{\text{true value}} \times 100\,\%$$

12.3 The matrix spike and analytical spike percent recovery calculation:
MS Percent Recovery:

$$\text{MS \% Recovery} = \left(\frac{\text{Observed Conc. of MS} - \text{Conc. of Smp}}{\text{True Spike Conc.}} \right) \times 100\ \%$$

12.3.1 When sample concentration is less than the instrument detection limit, use 0 for sample results only for purposes of calculating percent recovery.

12.3.2 The units for reporting spike sample results will be identical to those used for reporting sample results (µg/L for aqueous and mg/Kg dry weight basis for solid).

12.4 Duplicate Sample Relative Percent Difference calculation:

$$RPD = \frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2}\right)} \times 100\ \%$$

Where:

X1 = Original Result
X2 = Duplicate Result

13. POLLUTION PREVENTION

13.1 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. WASTE MANAGMENT

14.1 The following waste streams are produced when this method is carried out.

14.1.1 Filter paper contaminated with bismuth, lead or cadmium sulfide. This waste is collected in waste containers identified as "Lab Trash with Metals", Waste #39.

14.1.2 Aqueous rinsates from distillation tube clean up. This waste is placed in containers identified as "Acid Waste", Waste #33. It is neutralized to a pH between 6 and 9 and disposed down a lab sink.

14.1.3 Unused sample distillates. This waste is placed in waste containers identified as "Sodium Hydroxide / Pyridine Waste", Waste #1.

14.1.4 Miscellaneous solid waste contaminated with sample residue, acids, caustics and reagents used in this SOP. This waste is placed in a trash container and disposed with regular trash.

14.1.5 Aqueous analytical waste, neutral to slightly basic, contaminated with 3% pyridine. This waste is placed in waste containers identified as "Sodium Hydroxide / Pyridine Waste", Waste #1.

14.1.6 Aqueous alkaline material from the auto-analyzer/titrations. This waste is placed in waste containers identified as "Sodium Hydroxide / Pyridine Waste", Waste #1.

15. REFERENCES

15.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition; Total and Amenable Cyanide, Automated UV; Method 9012 A, Revision 1, December 1996.

15.2 EPA 600/4-79-020, Methods for Chemical Analysis of Water and Wastes, March 1983; Cyanide, Total; Method 335.4.

15.3 PITT-QA-DoD-0001, Implementation of the DoD QSM Version 3.

15.4 PITT-QA-0024, Subsampling.

16. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

16.1 All sample preparation and analysis information will be documented. Raw data will be forwarded for reporting and for inclusion in the project files.

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OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: Acid Volatile Sulfides (AVS) and Simultaneously Extracted Metals (SEM) in Sediment

(SUPERSEDES: None)

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1. SCOPE AND APPLICATION

1.1. This method describes for the determination of acid volatile sulfides (AVS) and for metals that are soluabilized duing the acidification step (Simultaneously Extracted Metals, SEM). The conditions used have been reported to measure amorphous or moderately crystalline monosulfides. As a precipitant of toxic heavy metals, sulfide is important in controlling the bioavailability of metals in anoxic sediments. If the molar ratio of toxic metals measured by SEM to AVS exceeds one, the metals are potentially bioavailable. Because the relative amounts of AVS and SEM are important in the prediction of potential metal bioavailability, it is important to use the SEM procedure for sample preparation for metal analysis. This uses the same conditions for release of both sulfide and metal from the sediment and thus provides the most predictive means of assessing the amount of metal associated with the sulfide.

1.2. Method 9034 is used to quantify the concentration of sulfide and Method 6010B is used to quantify the concentration of the routine SEM metals (cadmium, copper, lead, nickel, and zinc). If mercury is requested as a SEM, Method 7470A is used for quantification.

2. SUMMARY OF METHOD

2.1. The AVS in the sample is first converted to hydrogen sulfide (H_2S) by acidification with hydrochloric acid at room temperature. The H_2S is then purged from the sample and trapped. The amount of sulfide that is trapped is then determined titrimetrically following Method 9034, The SEM are metals liberated from the sediment during the acidification. These are determined following Method 6010B after filtration of the sample (plus 7470A if mercury is required).

3. DEFINITIONS

3.1. ACID VOLATILE SULFIDES (AVS): Amorphous, moderately crystalline monosulfides, and other sulfides that form hydrogen sulfide under the conditions of this test.

3.2. SIMULTANEOUSLY EXTRACTED METALS (SEM): Metals which form less soluable sulfides than do iron or manganese, and which are at least partially soluable under the conditions of this test. The routine SEMs are cadmium, copper, lead, nickel, and zinc. Mercury may also be determined on a project specific basis.

3.3. ICV: Initial Calibration Verification: An undistilled standard prepared by adding 1 mL of a 1000 ppm (or standardized concentration) sodium sulfide standard (different source than the standard used for the LCS and MS/MSD) to 50 mL of reagent water (20 ppm

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concentration).

3.4. ICB: Initial Calibration Blank: undistilled blank consisting of 50 mL of reagent water.

3.5. PBW: Prep Blank Water or Method Blank.

3.6. CCV: Continuing Calibration Verification : preparation is the same as the ICV.

3.7. CCB: Continued Calibration Blank: preparation is the same as the ICB.

3.8. LCS: Laboratory Control Sample.

4. INTERFERENCES

4.1. Oxygen in the reagents and apparatus is the primary interference reported. Samples must be taken with minimum aeration to avoid volatilization of sulfide or reaction with oxygen, which oxidizes sulfide to sulfur compounds that are not detected. Use deoxygenated, deionized water and reagents.

4.2. Reduced sulfur compounds, such as sulfite and hydrosulfite, may decompose in acid and form sulfur dioxide. This gas may carry over to the zinc acetate solutions and subsequently react with iodine during the titration, thus causing a positive bias to the results. Addition of formaldehyde to the zinc acetate scrubber removes this interference. Any sulfur dioxide entering the scrubber will form an addition compound with the formaldehyde which is unreactive towards the iodine in the acidified mixture.

4.3. The iodometric method suffers interference from reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds.

4.4. The pH of the sample after the addition of the acid and during the purge process must be below 3.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

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5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor or an STL Emergency Coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Boiling tube.

6.2. Inlet adapter.

6.3. Dropping funnel.

6.4. Gas inlet.

6.5. Impinged bubbler.

6.6. Fritted bubbler.

6.7. Bubbler vessels.

6.8. WestClips®

6.9. Gas line “T” connector.

6.10. Class A Volumetric flasks, pipets, and burets.

6.11. High purity nitrogen gas.

6.12. Regulator.

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6.13. 100 mL and 300 mL graduated disposable flasks.

6.14. 100 mL disposable beaker

6.15. Hot plate stirrer.

6.16. 50mL buret.

6.17. Parafilm

6.18. Filtering apparatus and 0.45 µm filter membrane.

7. REAGENTS AND STANDARDS

7.1. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee of 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.

7.2. Reagent water (Super Q/DI Water). All references to water in this method refer to reagent water.

7.3. Formaldehyde (37% solution), CH_2O. This solution is commercially available.

7.4. Zinc acetate for the scrubber. Zinc acetate solution (approximately 0.5M). Dissolve about 110g zinc acetate dihydrate in 200mL of reagent water. Add 1mL hydrochloric acid (concentrated), HCL, to prevent precipitation of zinc hydroxide. Dilute to 1L.

7.5. Acid to acidify the sample. 6 M Hydrochloric acid, 1:1 HCl:reagent water. Purge with nitrogen for at least 30 minutes prior to use.

7.6. 1:4 Sulfuric Acid (H_2SO_4): Carefully add 250 mls of concentrated sulfuric acid to a 1 liter volumetric flask containing at least 500 mls of reagent water. Bring up to volume.

7.7. UHP/zero grade nitrogen gas. Gas chromatographic grade with two-stage regulator.

7.8. Starch indicator. 0.5%. Purchased.

7.9. 0.0250N $Na_2S_2O_3$. Purchased.

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7.10. 0.025N Iodine. Purchased.

7.11. 1000ppm Sodium sulfide prepared by adding 3.75g $Na_2S \bullet 9H_20$ to 500mL reagent water. May be commercially available.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples must be cooled to 4^OC and stored headspace free. Typically a separate 4 oz jar is filled specifically for this test.

8.2. The acidification of the sample (H_2S generation) and sulfide determination must be performed within 14 days from the date of collection. The routine SEMs are stable up to six months after sample collection (28 days for mercury, if required).

9. QUALITY CONTROL

9.1. All quality control data must be maintained and available for reference or inspection for a period of three years. This method is restricted to use by or under supervision of experienced analysts.

9.2. A sulfide run will consist of the following sequence: ICV, ICB, and up to 10 samples followed by a CCV and a CCB. See the appropriate metals SOP for the SEM analyses

9.3. This can be followed by up to 10 more samples, followed by a CCV and CCB.

9.4. Repeat 9.2 and 9.3 sequence for additional samples.

9.5. A Laboratory Control Sample (LCS) must be analyzed with each batch of 20 or fewer samples. A separate sulfide (AVS) LCS and metals (SEM) LCS is performed.

9.6. A matrix spike and a matrix spike duplicate (MS/MSD) must be analyzed with each batch of 20 or fewer samples. A separate sulfide (AVS) MS/MSD and metals (SEM) MS/MSD is performed.

9.7. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in STL QA Policy: QA-005. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL reporting limit.

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9.8. A method blank must be analyzed with each batch of 20 samples or fewer processed at the same time. The prep blank (or method blank) can be used as the ICB if it meets the ICB acceptance criteria. The processing of a method blank will assure non-contamination of the reagents. The ICB result must be less than the REPORTING LIMIT. The method blank result must be less than two times the REPORTING LIMIT, otherwise all samples must be reprepared and reanalyzed. If this is not possible due to limited sample quantity (or there is no sample left) the corresponding samples will be flagged and the PM will be notified. If repreparation and reanalysis happen to be outside holding time, then approval from the client must be obtained before any reanalysis is performed.

9.9. The following QC requirements must be met for the sulfide (AVS) analyses:

9.9.1. The ICV must be within ± 15 percent. If this criteria is not met, then recalibrate and reanalyze the samples. The LCS can be used as an ICV if it meets the ICV acceptance criteria of 85 to 115 percent. If the LCS is not used as the ICV, then the LCS must meet a 75 to 125 percent recovery criteria.

9.9.2. The CCV must be within ± 15 percent. If this criteria is not met, then reanalyze the samples with a valid CCV. If the analysis sequence shows ICV, ICB, and 10 samples followed by CCV, CCB, and this CCV fails, then all those 10 samples must be reanalyzed. If with the above sequence 10 additional samples are analyzed following a CCV and a CCB and this second CCV fails, then all the samples up to the last acceptable CCV must be reanalyzed. The CCB criteria is the same as ICB.

9.9.3. The percent recovery for matrix spike and matrix spike duplicate should be ± 25 percent. If this criteria is not met, evaluate method process. If no errors are found, document in a Non-Conformance Memo (NCM).

9.9.4. The relative percent difference (RPD) between the MS and MSD must be within ± 20 percent. If this criteria is not met, then repeat the analysis once. The results with the better RPD will be reported. If the results for the reanalysis is the same as the original analysis, then report the original analysis.

9.10. Stock sulfide standard is titrated daily before each distillation of sample sets. The stock standard must be reprepared every week.

10. CALIBRATION AND STANDARDIZATION

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NOTE: All periodic standardizations of titrants can be found in the Wet Chemistry standardization log book. Daily standardizations are found on the bench worksheet.

10.1. Sodium thiosulfate (0.0250N) standardization—performed daily.

10.2. Use 0.0250 N Biiodate titrant: dissolve 0.8124g potassium biiodate (dried 2 hours) in Super-Q water and dilute to 1 L.

10.3. Place 2g KI in 250mL beaker and add 100mL Super-Q water and stir. Add 10mL 1:4 H_2SO_4 and 10mL biiodate.

10.4. Place in the dark for 5 minutes. Dilute to 150mL and add starch indicator (Section 7.7)

10.5. Titrate with $Na_2S_2O_3$ (7.8) to clear endpoint. Repeat procedure two additional times. Determine the normality of the $Na_2S_2O_3$ as follows:

$$N\ Na_2S_2O_3 = \frac{10\ mL\ biiodate \times 0.025\ N\ biiodate}{mL\ Na_2S_2O_3\ titrant}$$

10.6. Iodine standardization: performed daily

10.6.1. Place 20mL .0250N iodine in Erlenmeyer flask. Add 2mL 6 N HCl.

10.6.2. Titrate with $Na_2\ S_2O_3$ (7.8) to a pale yellow color.

10.6.3. Add starch indicator (7.7) and titrate with $Na_2S_2O_3$ (7.8) to clear endpoint. Determine the normality of the iodine (I) as follows:

$$N\ I = \frac{N Na_2S_2O_3 \times mL\ Na_2S_2O_3\ titrant}{mL\ I\ solution}$$

11. PROCEDURE

11.1. Place the boiling tube containing approximately 10 grams of sample (record to the nearest 0.1 grams) and 100 mL of reagent water in the heater block (used as a holder only) and assemble the acid soluble sulfide distillation apparatus as shown in Figure 1. The sample can be weighed on a 2” x 2” piece of Parafilm and placed into the boiling tube.

11.2. Spike the sulfide (AVS) LCS, MS, and MSD with 1 mL of the 1000 ppm sodium sulfide solution (7.10) which is equivalent to 100 mg/Kg in a 10 gram sample. Spike the metals

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(SEM) LCS, MS, and MSD with 2.5 mLs of the metals ICP MS solution. If mercury is required, a mercury spike will need to be added to the LCS, MS, and MSD.

11.3. Place 2.0mL of 0.5M zinc acetate solution, 20.0mL of deionized water, and 1.0mL of 37% formaldehyde in each of two bubbler vessels. Place an impinged bubbler in the first (front) and second (back) vessel, and seal them with size 24/40 WestClips®. The sealed vessels and impingers function as the gas scrubbers. Connect the first scrubber to the inlet adapter and place the second bubbler vessel in the bubbler vessel rack. Connect the two impingers in series using Tygon® tubing.

11.4. Close stopcock of dropping funnel. Place 20 mL of the nitrogen purged 6 M hydrochloric acid in the dropping funnel.

11.5. Connect a high-purity (GC grade) nitrogen gas source to the main inlet of the gas manifold of the aluminum heater block as specified in the Heater Block Operation Manual. Use a two-stage gas tank regulator and set the pressure into the gas manifold to 20psi.

11.6. Connect a black gas line from each gas manifold valve to a "T" connector and a tygon gas line from the "T" to each of the two gas inlets of the apparatus. One at the top of the dropping funnel and one at the inlet adapter as shown in **Figure 1**.

11.7. Purge assembled apparatus with high-purity nitrogen for 10 minutes to remove atmospheric oxygen from the apparatus and contained solutions. During purge, adjust nitrogen flow such that 2-3 bubbles per second exit the base of the inlet adapter.

11.8. Open stopcock of dropping funnel and allow all of the 6M hydrochloric acid to drip into the boiling tube. Once dropping funnel is empty, close the stopcock to ensure sample is not lost into the funnel.

11.9. Purge the sample for 1 hour at room temperature. After the 1 hour purging period, remove the bubbler vessels. Turn off the nitrogen flow. Carefully combine the gas scrubber solutions in a 100 mL graduated disposable flask. Do not shake or mix solutions to avoid loss of sulfide. Bring up to 50 mL with reagent water. Determine the concentration of acid volatile sulfide in the zinc acetate gas scrubber solutions by using the Titrimetric-iodine method (9034)—proceed to Section 11.11.

11.10. After the generation of sulfide has been completed, the sediment suspension remaining in the boiling tube is filtered through a 0.45 µm membrane filter. The pH of the solution is determined using narrow range pH strips to verify that the pH is less than 3. If the pH is not less than 3, the group supervisor and QA Manager should be consulted. Document all actions in a Nonconformance Memo (NCM). The solution is brought up to a final volume

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of 250 mL in a 300 mL graduated disposable flask. This solution is analyzed directly by ICP for the routine SEMs (see SOP C-MT-0001). If mercury is required, an aliquot of this solution is prepared following Method 7470A and analyzed by CVAA (see SOP C-MT-0005).

11.11. Pipet a known amount of standardized 0.025N iodine solution in a 100mL disposable beaker, adding an amount in excess of that needed to oxidize the sulfide.

11.12. Add 2mL of 6N HCl to the iodine.

11.13. Pour the gas scrubbing solutions obtained in Section 11.9 to the flask. If at any point in transferring the zinc acetate solution, the amber color of the iodine disappears or fades to yellow, more 0.025N iodine must be added. This additional amount must be added to the amount from Section 11.11 for calculations. Record the total volume of standardized 0.025N iodine solution used.

11.14. Titrate the solution in the flask with standard 0.025N sodium thiosulfate solution until the amber color fades to yellow. Add enough starch indicator for the solution to turn dark blue and titrate until the blue disappears. Record the volume of titrant used.

12. DATA ANALYSIS AND CALCULATIONS

12.1. One mL of 0.0250 N standard iodine solution reacts with 0.4mg sulfide present in titration vessel.

12.2. $$\text{AVS mg/Kg-dry} = \frac{[(A \times B) - (C \times D)] \times 16000}{E \times F}$$

A = mL of iodine solution
B = N of iodine solution
C = mL of $Na_2S_2O_3$ solution
D = N of $Na_2S_2O_3$ solution
E = weight of sample (grams)
F = Percent solids as decimal fraction (i.e., 50% solid is 0.50)

12.3. To convert the AVS concentration from mg/Kg-dry to µmoles/gram-dry, divide by 32.066 (molecular weight of sulfur).

12.4. Enter the completed data work sheet into computer program, sulfide analysis worksheet, for final results.

12.5. For each SEM, first determine concentration in mg/Kg-dry as follows:

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$$\text{SEM mg/Kg-dry} = \frac{A \times B}{C \times D}$$

A = conc. of metal in solution as determined by 6010B or 7470A (mg/L)
B = final volume of solution in liters—typically 0.25 liters.
C = weight of sample in Kg.
D = Percent solids as decimal fraction (i.e., 50% solid is 0.50)

12.6. To convert the concentration of each SEM from mg/Kg-dry to µmoles/gram-dry, divide by the molecular weight of that metal (cadmium = 112.411; copper = 63.546; lead = 207.2; mercury = 200.59; nickel = 58.69; and zinc = 65.39).

12.7. Calculate the Total SEM molar concentration of the sample by summing each of the individual SEM concentrations in units of µmoles/gram-dry. If any one of the SEMs is not detected (ND), it is considered a zero (0) in the summation.

12.8. Calculate the molar ratio of SEM over AVS as follows:

SEM/AVS = A/B

A = Total SEM molar concentration (µmoles/gram-dry).
B = AVS molar concentration (µmoles/gram-dry).

Note: If AVS is not detected (ND), the molar ratio cannot be determined.

12.9. Matrix Spike percent recovery:

$$Theoretical\ Spike\ Conc. = \frac{\left(\begin{matrix}Spike\\Conc.\end{matrix}\right) \times \left(\begin{matrix}Vol.\ of\\Spike\ Added\end{matrix}\right)}{Final\ Vol.\ Spiked}$$

$$\%\ \text{Recovery} = \frac{\left(\begin{matrix}Final\\Spike + Sample\end{matrix}\right) - Sample\ \text{Result}\left(\begin{matrix}Vol.\ Sample\\Spiked\\ \hline Final\ Vol.\\Spiked\end{matrix}\right)}{Theoretical\ Spike\ Conc.} \times 100$$

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13. METHOD PERFORMANCE

13.1. Training Qualifications

13.2. The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. The group/team leader must document the training and PE performance and submit the results to the QA Manager for inclusion in associate training files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure will be segregated, and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Coordinator should be contracted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3nd ed.; U.S. EPA. Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1997; SW-846.

16.2. Allen, H.E. and F. Gongmin et al. 1991. Determination of Acid Volatile Sulfide and Simultaneously Extractable Metals in Sediment, April 1991 (Draft Analytical Method for the Determination of Acid Volatile Sulfide in Sediment, U.S. EPA Office of Water and Office of Science and Technology, Health and Ecological Criteria Division, Washington, D.C., August 1991

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. All sample preparation and analysis information will be documented on laboratory bench sheets, computer printouts, standard logbooks, etc. All the documents associated with an analysis will be forwarded for reporting and for inclusion in the project files.

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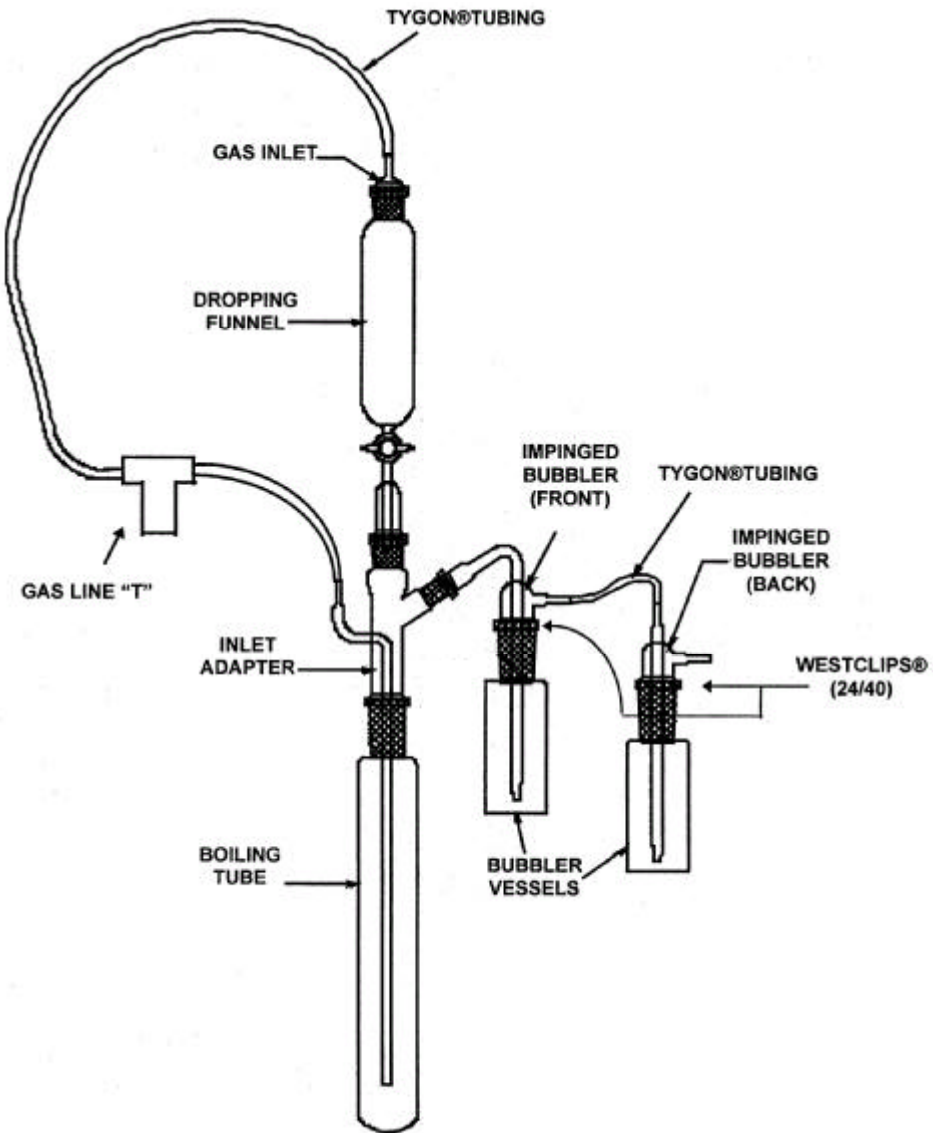

Figure 1 – Acid Volatile Sulfide generation apparatus.

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Title: Gas Chromatographic Analysis

Method(s): SW-846 Methods 8000B, 8081A, 8082, 8141A, 8151A, 8310, 8041 and EPA Method 610

Approvals (Signature/Date):

9/26/07	9/20/07
John Oravec Date	Larry Matko Date
Technical Manager	Acting Health & Safety Manager
9/20/07	9/20/07
Nasreen DeRubeis Date	Larry Matko Date
Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. PITT-GC-0001, Rev. 11.

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1 SCOPE AND APPLICATION

This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC) and High Performance Liquid Chromatography (HPLC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices.

2 SUMMARY OF METHOD

In general, semivolatile analytes in aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction (SOP # PITT-OP-0001). Solid samples are prepared using sonication, soxhlet or accelerated soxhlet extraction (SOP # PITT-OP-0001).

After the initial preparation step, the sample is introduced to the GC or HPLC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3 DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the Laboratory Quality Assurance Plan (LQM).

4 INTERFERENCES

Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors. See the appendices for interferences specific to individual tests and suggested corrective actions.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 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.

5.3 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.

5.4 The following method parameters have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzo(a)pyrene, and dibenzo(a,h)-anthracene.

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5.5 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.
Acetonitrile	Flammable Poison	40 ppm-TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
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.

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5.6 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.7 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported immediately to a laboratory supervisor or the EH&S coordinator.

6 EQUIPMENT AND SUPPLIES

An analytical system complete with a gas chromatograph or high performance liquid chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7 REAGENTS AND STANDARDS

7.1 Stock Standards

7.2 Stock standards are purchased as certified solutions or prepared from pure solutions. Semivolatile stock standard solutions are stored at 4.0 °C ± 2°C. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

7.3 Semivolatile stock standard solutions must be replaced after one year.

7.4 Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If a vendor supplied standard has an earlier expiration date then that date is used.

7.5 Calibration Standards

7.6 Semivolatile Calibration Standards: Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at 4.0 °C ± 2°C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem. Refer to each appendix for the preparation of calibration standards. **For Method 8141A the standards are to be replaced after 2 months.**

7.6.1.1 A minimum of a five-point calibration curve is prepared from commercially purchased custom standards. A proprietary software database program is utilized for providing and managing all necessary detail regarding standards associated with this method. An example copy of a print out available from this database is provided in Figure 1. Refer to each Appendix for a summary of standard preparation of the calibration standards. The lowest calibration point must be at or below the reporting limit.

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7.7 Gases for carrier and make-up: Hydrogen, Helium, Nitrogen, Argon/Methane.

7.8 Quality control (QC) Standards

7.9 QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

8 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Semivolatile extracts must be refrigerated at 4.0 °C ± 2°C and analyzed within 40 days of the end of the extraction. Extracts for methods 610/8310/8041 must be protected from light and refrigerated at 4.0 °C ± 2°C.

9 QUALITY CONTROL

9.1 Refer to the TestAmerica Pittsburgh QC Program document (QA-003) for further details on criteria and corrective actions. Refer to “Project Checklist” for project specific requirements.

9.2 For specific DoD quality control requirements refer to SOP # PITT-QA-DoD-0001, Implementation of the DoD QSM Version 3, January 2006 and Appendix H in this SOP.

9.3 Initial Demonstration of Capability

9.4 For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin.

9.5 For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.6 Batch Definition

9.7 Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica QC Program document (QA-003) for further details of the batch definition.

9.8 Quality Control Batch: The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD a duplicate LCS (LCSD) may be

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substituted. (For method 610, a blank, LCS and matrix spike must be performed every 10 samples.)

9.9 Control Limits: For DoD work the control limits are listed in the SOP PITT-QA-DoD-0001. For DoD QC acceptance criteria refer to DoD SOP listed above. In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be verified at least annually. The recovery limits are mean recovery +/- 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Refer to policy QA-003 for more details.

9.10 These limits do not apply to dilutions, but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.11 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated.

9.12 Refer to the QC Program document (QA-003) for further details of control limits.

9.13 Surrogates: All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

9.14 Check all calculations for error.

9.15 Ensure that instrument performance is acceptable.

9.16 Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.

9.17 Reprepare and reanalyze the sample or flag the data as “Estimated Concentration” if neither of the above resolves the problem. Repreparation is not necessary is there is obvious chromatographic interference.

9.18 The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.19 The Surrogate in the method blank and LCS must perform within limits. The exception is where the surrogates are out high and all the samples are non-detected. For all other cases the batch must be reprepped and re-analyzed. Regardless, whenever a surrogate is outside of limits in a blank and LCS corrective action should be taken to identify and correct the problem.

9.20 If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in-control result is reported.

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9.21 If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

9.22 Refer to the TestAmerica QC Program document (QA-003) for further details of the corrective actions.

9.23 Method Blanks

9.24 For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatiles samples, and sodium sulfate for semivolatile solids tests (Refer to SOP No. PITT-OP-0001 for details).

9.25 Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher. Wherever blank contamination is greater than 1/10 the concentrations found in the samples and/or 1/10 of the regulatory limit it is potentially at a level of concern and should be handled as a non-conformance. Blank contamination should always be assessed against project specific requirements (See associated project checklist).

9.26 If the analyte is a common laboratory contaminant (ex. phthalate esters) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

9.27 Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.28 If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.29 Instrument Blanks

9.30 An instrument blank consists of the appropriate solvent with internal standards added. If internal standards are not used the surrogates should be added.

9.31 An instrument blank may be analyzed after calibration standards or suspected high concentration samples to ensure that there is no carryover.

9.32 Laboratory Control Samples (LCS)

9.33 For each batch of samples, analyze a LCS. The LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action

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must occur. Corrective action will normally be repreparation and reanalysis of the batch.

9.34 Refer to the TestAmerica QC Program document (QA-003) for further details of the corrective action.

9.35 If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 11.1.2.1) unless one column is out of control, in which case the in control result is reported.

9.36 LCS compound lists are included in each of the appendices.

9.37 If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client.

9.38 Matrix Spikes

9.39 For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits.

9.40 If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.

9.41 If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.

9.42 If a MS/MSD is not possible due to limited sample, then a LCS duplicate (LCSD) should be analyzed.

9.43 The MS/MSD must be analyzed at the same dilution as the unspiked sample.

9.44 If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 11.1.2.1) unless one column is out of control, in which case the in control result is reported.

9.45 Quality Assurance Summaries

9.46 Certain clients may require specific project or program QC, which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

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9.47 TestAmerica QC Program

9.48 Further details of QC and corrective action guidelines are presented in the TestAmerica QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10 PROCEDURE

10.1 Calibration and Standardization

10.2 Internal or external calibration may be used. In either event prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard must be at or below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

10.3 For specific DoD calibration requirements refer to SOP # PITT-QA-DoD-0001, Implementation of the DoD QSM Version 3, January 2006.

10.4 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance.

10.5 With the exception of 10.1.5 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Third order calibrations require at least seven points.

10.6 A level may be removed from the calibration if the reason can be clearly documented, for example a broken vial. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.7 External standard calibration

Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC or HPLC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$Calibration\ Factor\ (CF) = \frac{Area\ or\ Height\ of\ Peak}{Mass\ Injected\ (ng)}$$

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Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. It is also possible to use the concentration of the standard rather than the mass injected. (This would require changes in the equations used to calculate the sample concentrations). Use of peak area or height must be consistent. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

10.8 Internal standard calibration

10.8.1.1 The internal standard approach assumes that variations in instrument sensitivity, amount injected etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. To use this approach, select one or more internal standard(s) that are similar in analytical behavior to the compounds of interest. Recommended internal standards are given in the appendices. The analyst must demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. If the sample matrix interferes with quantitation of the internal standard, then the external standard approach must be used instead. In this event use the response factors from the previous continuing calibration to quantitate the analytes in the sample with the interference (applies only to the sample with the interference).

10.8.1.2 Introduce each calibration standard into the GC or HPLC using the technique that will be used for samples. Response factors (RF) for each compound are calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:
A_S = Response for the analyte to be measured
A_{IS} = Response for the internal standard
C_{IS} = Concentration of internal standard

C_S = Concentration of the analyte to be determined in the standard

10.8.2 Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the % RSD ≤ 20% (see equation below for the calculation of %RSD for each compound in curve).

$$\%RSD = \frac{SD}{RRF_A} x100$$

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Where:

%RSD = Percent Relative Standard Deviation

SD = Standard Deviation (n-1) of the response factors or calibration factors in curve

RRF_A = Average response factor or calibration factor of all points in curve

10.8.2.1 In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector.

10.8.2.2 Average response factor (or calibration factor)

The average response factor may be used if the average percent relative standard deviation (%RSD) is ≤ 20%. For method 610 the % RSD must be < 10%.

The equation for average response factor is:

$$Average\ response\ factor = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.8.2.3 Linear regression

The linear fit uses the following functions:

10.8.2.3.1 External Standard

$$y = ax + b$$

or

$$x = \frac{(y - b)}{a}$$

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Where:

y = Instrument response

x = Concentration

a = Slope

b = Intercept

10.8.2.3.2 Internal Standard

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

Where:

C_s = Concentration in the sample

A_s= Area of target peak in the sample

A_{is}= Area of internal standard in the sample

C_{is} = Concentration of the internal standard

10.8.2.4 Quadratic curve

The quadratic curve uses the following functions:

10.8.2.4.1 External standard

$$y = ax + cx^2 + b$$

Where:

C = Curvature

10.8.2.4.2 Internal Standard

$$y = a\left(\frac{A_s \times C_{is}}{A_{is}}\right) + c\left(\frac{A_s \times C_{is}}{A_{is}}\right)^2 + b$$

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Note: Quadratic curve is not used for any South Carolina samples.

10.8.3 Evaluation of calibration curves

10.8.3.1 The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response must be less than ± the reporting limit for the analyte.
- The Percent Relative Standard Deviation (%RSD) for average response factors or calibration factors for all analytes in the calibration standards used must be ≤ 20%. For method 610 the % RSD must be < 10%.
- For linear regression calibrations, the Correlation Coefficient (*r*) is used, and must be greater than or equal to 0.990.
- For the quadratic calibrations, the Coefficient of Determination (r^2) is be used, and must be greater or equal to 0.990.
- For multiple component analytes, each peak used in quantitating the compound should have a %RSD that is ≤ 20%.

10.8.4 Weighting of data points

10.8.4.1 In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/Concentration^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.8.5 Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation.

10.8.6 Calibration Verification

10.8.6.1 Initial Calibration Verification (ICV) (Second Source)

Each Initial Calibration (ICAL) will be verified prior to sample analyses by the analysis of a second-source calibration standard (ICV). This standard must be prepared from a source(s) that is independent from the ICAL standards. **The ICV acceptance criteria is ± 20% of expected value (QSM requirement also.)**

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10.8.6.2 Continuing Calibration Verification (CCV)

It may be appropriate to analyze a mid point standard more frequently than every 12 hours. If these calibration verification standards are analyzed, requirements are the same as the 12 hour calibration with the exception that retention times are not updated. To meet the NELAC requirement for verification of the ICAL at varied concentrations, a CCV, at a concentration other than the mid point concentration, will be analyzed daily. The continuing calibration verification requirements for DoD work is listed in SOP PITT-QA-DoD-0001.

10.8.6.3 Any individual compounds with a %D $\leq$ 15% meet the calibration criteria. For multiple component compounds, each peak used to quantitate the compound must have a %D $\leq$ 15%.

10.8.6.4 When the acceptance criteria for the continuing 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. (This is according to NELAC section 5.5.5.8.1 June 5,2003.)

10.8.6.5 It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration and an acceptable ICV.

10.8.6.6 Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above. Bracketing is not necessary for internal standard methods.

10.8.6.7 If the analyst notes that a CCV has failed and can document the reason for failure (e.g. broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then the preceding samples have been successfully bracketed. If adjustments to the instrument are performed before the repeat CCV then the preceding samples have not been successfully bracketed but analysis may continue.

10.8.6.8 In general, it is not advisable to analyze repeat CCVs on unattended runs. If repeat CCVs are analyzed then the first will serve as the bracketing standard for the preceding samples and the last will serve as the CCV for the following samples. **The ending CCV must pass criteria**, see 10.11.3-10.11.4.

10.8.6.9 If highly contaminated samples are expected it is acceptable to analyze blanks or primers at any point in the run.

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10.8.6.10 % Difference calculation

% Difference for internal and external methods is calculated as follows

Internal Standard:

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100$$

External standard:

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where:

RF and CF are the response to calibration factors from continuing calibration

$\overline{RF}$ and $\overline{CF}$ are the average response and calibration factors from the initial calibration

10.8.6.11 % Drift calculation

% Drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for % Drift are the same as for % Difference

$$\%\ Drift\ = \frac{Calculated\ Conc.\ -\ Theoretical\ Conc.}{Theoretical\ Conc.} \times 100\%$$

10.8.6.12 Corrective Actions for Continuing Calibration

If the %D of any analyte is greater than ± 15% corrective action must be taken. . Where there is indication that the instrument ongoing performance has been altered corrective action may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. . If the %D exceeds reportable ranges and cannot be corrected by instrument maintenance a new calibration curve must be prepared.

10.8.6.13 Corrective Action for Samples

For internal standard methods, any samples injected after a standard not meeting the calibration criteria must be reinjected.

For external standard methods, any samples injected after the last good continuing calibration standard must be reinjected.

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10.9 Procedure

10.9.1 For specific DoD requirements refer to SOP # PITT-QA-DoD-0001, Implementation of the DoD QSM Version 3, January 2006.

10.9.2 Extraction

10.9.2.1 Extraction procedures are referenced in SOP PITT-OP-0001.

10.9.3 Cleanup

10.9.3.1 Cleanup procedures are referenced in SOP PITT-OP-0001.

10.9.3.2 Carboprep 90 Cleanup

10.9.3.3 This cleanup may be performed prior to analyses for pesticides and/or PCBs (by methods 8081A or 8082) if the sample extract has some color.

10.9.3.3.1 Cartridge Method

10.9.3.3.1.1 Put approximately 2 ml of sample extract into a test tube and mark the sample volume on the tube.

10.9.3.3.1.2 Condition the cartridge by adding 2 ml of methylene chloride and allowing it to drip through the cartridge. Do not allow the cartridge packing to go dry in this or any subsequent step, until the final rinse has been completed.

10.9.3.3.1.3 Add 2 ml of hexane/methylene chloride (80%/20%) mixture and allow it to drip through the cartridge until almost empty.

10.9.3.3.1.4 Add the sample extract to the cartridge and place the test tube under the cartridge to collect the liquid as it drips through.

10.9.3.3.1.5 Rinse 3 times with 2 ml aliquots of hexane/methylene chloride (80%/20%) mixture, while not allowing the cartridge to go dry. After the final rinse, use a pipette bulb to force out all of the remaining liquid in the cartridge.

10.9.3.3.1.6 Concentrate the sample extract back down to the original volume according to the mark on the test tube. The extract is now ready for analysis.

10.9.3.3.2 Quick Method

10.9.3.3.2.1 Add a half scoop of Carboprep 90 to approximately 2 ml of sample extract.

10.9.3.3.2.2 Shake for one minute and allow the extract to settle.

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10.9.3.3.2.3 Pipette out an aliquot of the clear extract for analysis.

10.9.4 Chromatography

10.9.4.1 Chromatographic conditions for individual methods are presented in the appendices.

10.9.5 Sample Introduction

10.9.5.1 Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

10.9.6 Analytical Sequence

10.9.6.1 An analytical sequence starts with an initial calibration or a daily calibration. Refer to the individual method appendices for method specific details of daily calibrations and analytical sequences.

10.9.6.2 The daily calibration includes analysis of standards containing all single response analytes and updating the retention time windows.

10.9.6.3 If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration.

Retention Time Windows

10.9.6.4 Absolute retention times are used for the identification of PCBs as Aroclors. However, in addition to retention times, peak patterns play a large role in the identification of Aroclors.

10.9.6.5 Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.

10.9.6.6 Before establishing windows, make sure the GC system is within optimum operating conditions. Make 3 injections of all standard mixtures throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

10.9.6.7 Record the retention time for 3 to 5 major peaks for both Aroclor 1016 and Aroclor 1260 to 3 decimal places. Calculate the mean and the standard deviation of the 3 absolute retention times.

10.9.6.8 If the standard deviation of the retention times for a target compound is 0.000 (no difference between the 3 retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

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10.9.6.9 The width of the retention time window is defined as ± 3x the standard deviation of the absolute retention times. If the default standard deviation is employed, the width of the window will be 0.03 minutes.

10.9.6.10 Daily Retention Time Windows: Establish the center of the retention time windows from the calibration verification 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. Retention time windows can be updated every 12 hours. However, they are usually only updated at the onset of a continuing calibration sequence or after maintenance has been performed.

10.9.6.11 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory and available for review.

10.9.6.12 The laboratory monitors the retention time for both surrogates throughout the analytical sequence.

10.9.6.13 Corrective Action for Retention Times

The retention times of all compounds in each continuing calibration must be within the retention time windows established by the 12 hour calibration. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window:

The retention time of that compound in the standard must be within a retention time range equal to twice the original window and,

No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

10.9.7 Percent Moisture

10.9.7.1 Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight.

10.9.8 Procedural Variations

10.9.8.1 Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also 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.

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11 CALCULATIONS / DATA REDUCTION

11.1 Qualitative Identification

11.2 Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if estimated results ("J" qualified) are required. Normally confirmation is required on a second column or detector (Method 610/8310), but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases GC/MS confirmation may be required. Client specific requirements may also define the need for second column confirmation and/or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column or detector, at a concentration greater than the reporting limit or MDL (if "J" qualified confirmation is required).

11.3 Dual column or detector quantitation

11.3.1.1 When results are confirmed using a second GC column of dissimilar stationary phase, the analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed. Unless otherwise specified in an approved project plan, the **higher** result should be reported. **For DoD QSM V3.0,** report the **higher of two confirmed** results unless overlapping peaks are causing erroneously high results, then report the nonaffected result and document in the case narrative.

11.3.2 If the Percent Difference (%D) between the response on the two columns or detectors is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. If the CCV % D is within ± 15% for one column and >15% but < 20% for the other column and the %D between the two columns is within 40%, the data is reported from the column with the CCV within ± 15%. %D between column or detector results is calculated using the following formula:

$$\%D = (|R1 - R2|/R3) \times 100$$

Where:

R1 = First column/detector result

R2 = Second column/detector result

R3 = R1 or R2, whichever is lower

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11.3.3 Multi-response Analytes

11.3.3.1 For multi-response analytes, the analyst should use the retention time window, but should rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

PCBs: In the 3-5 peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of multi-component in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 3-5 resulting concentrations are averaged to provide the final result for the sample.

Toxaphene: In the 4-peak approach, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of Toxaphene in the standard. A minimum of three peaks must be used to determine the calibration factor. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 4 resulting concentrations are averaged to provide the final result for the sample.

Chlordane – Technical chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. The following components are significant: a- and g-chlordane, trans-nonachlor, heptachlor, and heptachlor epoxide. The a- and g-chlordane isomers are the most prevalent and their detection as single components is a good indicator that technical chlordane may be present. When the GC pattern of the residue resembles that of the technical chlordane standard, quantitate chlordane residues by comparing the area of 4 to 5 major peaks. Heptachlor and heptachlor epoxide should not be included in this quantitation but rather should be quantitated and reported separately.

11.3.3.2 The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

11.3.3.3 Equation for calculation of analyte response factor:

$$RF = \frac{Peak\ Area\ or\ Height\ in\ Standard}{Total\ Mass\ (ng)\ of\ Standard\ Inj.}$$

11.3.3.4 Equation for analyte concentration in aqueous samples:

$$Conc.\ ug/L = \frac{(Ax)(DF)(V_t)}{(RF)(V_I)\ (V_S)}$$

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Where

A_x = Area or height of the peak for the analyte in the sample.

V_t = Total volume of the concentrated extract (uL).

DF = Dilution factor. (DF = 1 for no dilution).

RF = Response Factor for single point or Mean Response Factor for multi-point curve.

V_I = Volume of extract injected in uL

V_s = Volume of aqueous sample extracted in mL

11.3.3.5 Equation for analyte concentration in soil samples:

$$Conc.\ ug/kg = \frac{(Ax)(DF)(V_t)}{(RF)(V_I)\ (W_S)}$$

Where

A_x = Area or height of the peak for the analyte in the sample.

V_t = Total volume of the concentrated extract (uL).

DF = Dilution factor. (DF = 1 for no dilution).

RF = Response Factor for single point or Mean Response Factor for multi-point curve.

V_I = Volume of extract injected in uL

W_s = Weight of sample extracted in gm

11.3.3.6 Note that the calibration and calculation of multi-point compounds is performed using the same equations for individual component peaks. However, in the case of multi-component compounds the individual component peak results are averaged for reporting the final result.

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11.4 Calibration Range

11.5 If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix interferences.

11.6 Dilutions

11.7 Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.8 Guidance for Dilutions Due to Matrix Interferences

11.9 If the sample is initially run at a dilution and only minor matrix interfering peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Dilutions 3-5X report the data and narrate. Dilutions greater than 5X then reported diluted out.

11.10 Reporting Dilutions

11.11 The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.12 Interferences

11.13 If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

11.14 Internal Standard Criteria for Continuing Calibration

11.15 If internal standard calibration is used, then the internal standard response in a continuing calibration standard must be within 50 to 150% of the response in the mid level of the initial calibration. The lab does not currently use internal standards for routine analysis.

11.16 Calculations

11.17 Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

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11.17.1 External Standard Calculations

11.17.1.1 Aqueous samples

$$Concentration\ (mg/L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample

V_i = Volume of extract injected, µL

D_f = Dilution factor

V_t = Volume of total extract, µL

V_s = Volume of sample extracted, mL

CF = Calibration factor, area or height/ng, Section 10.1

11.17.1.2 Non-aqueous Samples

$$Concentration\ (mg/kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

W = Weight of sample extracted, g

$$D = \frac{100 - \%\,Moisture}{100}$$

(D = 1 if wet weight is required)

11.17.2 Internal Standard Calculations

11.17.2.1 Aqueous Samples

$$Concentration(mg/L) = \frac{(Ax \times Cis \times Df \times Vi)}{(Ais \times \overline{RF} \times Vs)(1000)}$$

Where:

C_{is} = Amount of internal standard added, ng

A_{is} = Response of the internal standard

RF = Mane response factor for analyte

A_x = Response for the analyte in the sample

V_i = Volume of extract injected, µL

D_f = Dilution factor

V_s = Volume of sample extracted, mL

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11.17.2.2 Non-aqueous Samples

$$Concentration\ (mg/kg) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

11.17.3 Surrogate Recovery

11.17.3.1 Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{Concentration\ (or\ amount)\ found}{Concentration\ (or\ amount)\ spiked} \times 100$$

11.17.4 LCS Recovery

11.17.4.1 Concentrations of each compound in the LCS are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. LCS recoveries are calculated using the following equation:

$$\%\ \text{Recovery} = \frac{Concentration\ (or\ amount)\ found}{Concentration\ (or\ amount)\ spiked} \times 100$$

11.17.5 MS/MSD Recovery and RPD

11.17.5.1 Concentrations of each compound in the MS and MSD are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. The MS and MSD recoveries and the RPD between the MS and MSD are calculated using the following equations:

$$\%\ Spike\ Recovery\ (\%R) = \frac{SSR - SR}{SA}\ x\ 100$$

where:

SSR = Spiked Sample Result

SR = Sample Result

SA = Spike Added

$$RPD = \frac{|Conc.\ 1 - Conc.\ 2|}{(Conc.\ 1 + Conc.\ 2)/2} \times 100$$

where:

Conc. 1 = MS Concentration

Conc. 2 = MSD Concentration

11.18 For manual integration practices refer to TestAmerica corporate SOP, S-Q-004, Acceptable Manual Integration Practices. For DoD and all other projects the following criteria must be met:

When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of manual integration (i.e., chromatograms of manually integrated peaks), and notation of rationale, date, and signature/initials of person performing manual integration operation (electronic signature is acceptable). DoD QSM, Version 3, Clarification 50 and 57.

Case Narrative. For DoD the case narrative shall provide: identification of **samples and analytes** for which manual integration was necessary. DoD QSM, Version 3, Appendix DoD-A.

12 METHOD PERFORMANCE

Method Detection Limit: Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte and verified. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP#: PITT-QA-007. MDLs are analyzed yearly for each analyte of interest.

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12.1 Initial Demonstration: Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. IDOC is performed for each new analyst.

12.2 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid level calibration.

12.3 Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

12.4 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.5 Training Qualification

12.6 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13 POLLUTION CONTROL

13.1 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.”

13.2 This method does not contain any specific modifications that serve to minimize or prevent pollution.

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14 WASTE MANAGEMENT

14.1 The following waste streams are produced when this method is carried out.

14.2 Acetonitrile and water from the HPLC. This waste is collected in a waste container identified as “Mixed Flammable Solvent Waste”, Waste #3.

14.3 Methylene Chloride in vials. This waste is placed in waste container identified as “Vials & Extracts”, Waste #7.

14.4 Flammable solvents in vials. This waste is placed in waste container identified as “Vials & Extracts”, Waste #7.

14.5 Waste flammable solvents. This waste is collected in a waste container identified as “Mixed Flammable Solvent Waste”, Waste #3.

14.6 Expired primary and working PCB standards. This waste is placed in a waste container identified as “PCB Standard Waste”, Waste #8.

15 REFERENCES

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Section 8000B.

15.2 SOP # PITT-QA-DoD-0001, Implementation of the DoD QSM Version 3, January 2006, current version.

15.3 SOP # S-Q-004, Acceptable Manual Integration Practices, current version.

15.4 SOP # PITT-QA-0024, Subsampling.

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16 ATTACHMENTS

16.1 Figure 1 – Initial Demonstration and MDL Flow Diagram

16.2 Figure 2 – Sample Analysis Flow Diagram

16.3 Figure 3 – Example Standards Preparation Logbook Page

16.4 Appendix A – Analysis of PCB Congeners Based on Method 8082

16.5 Appendix B – Analysis of Organochlorine Pesticides Based on Method 8081A

16.6 Appendix C – Analysis of PCB Aroclors Based on Method 8082

16.7 Appendix D – Analysis of Organophosphorus Pesticides Based on Method 8141A

16.8 Appendix E – Analysis of PAHs Based on Methods 610 and 8310

16.9 Appendix F – Analysis of Phenols Based on Method 8041

16.10 Appendix G – Analysis of Herbicides Based on Method 8151A

16.11 Table H-1: Summary of QC Check Definitions, Purpose and Evaluation – Organics (GC/HPLC)

16.12 Table H-2: Organic Analysis by GC and HPLC – Methods 8081, 8082, 8141, 8151 and 8310

17 REVISION HISTORY

17.1 Revision 12, 09/07/2007

17.2 Corrected the text in Section 11.1.2.1 to read that whenever results are confirmed using a second GC column of dissimilar stationary phase, the **higher** of the two results should be reported.

17.3 Changed the format of the SOP to correspond to the new Corporate SOP format.

17.4 See highlighted areas within the SOP for changes.

18 METHOD MODIFICATIONS

18.1 Modifications from Reference Method

18.2 Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

Figure 1 - Initial Demonstration and MDL Flow Diagram

(This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.)

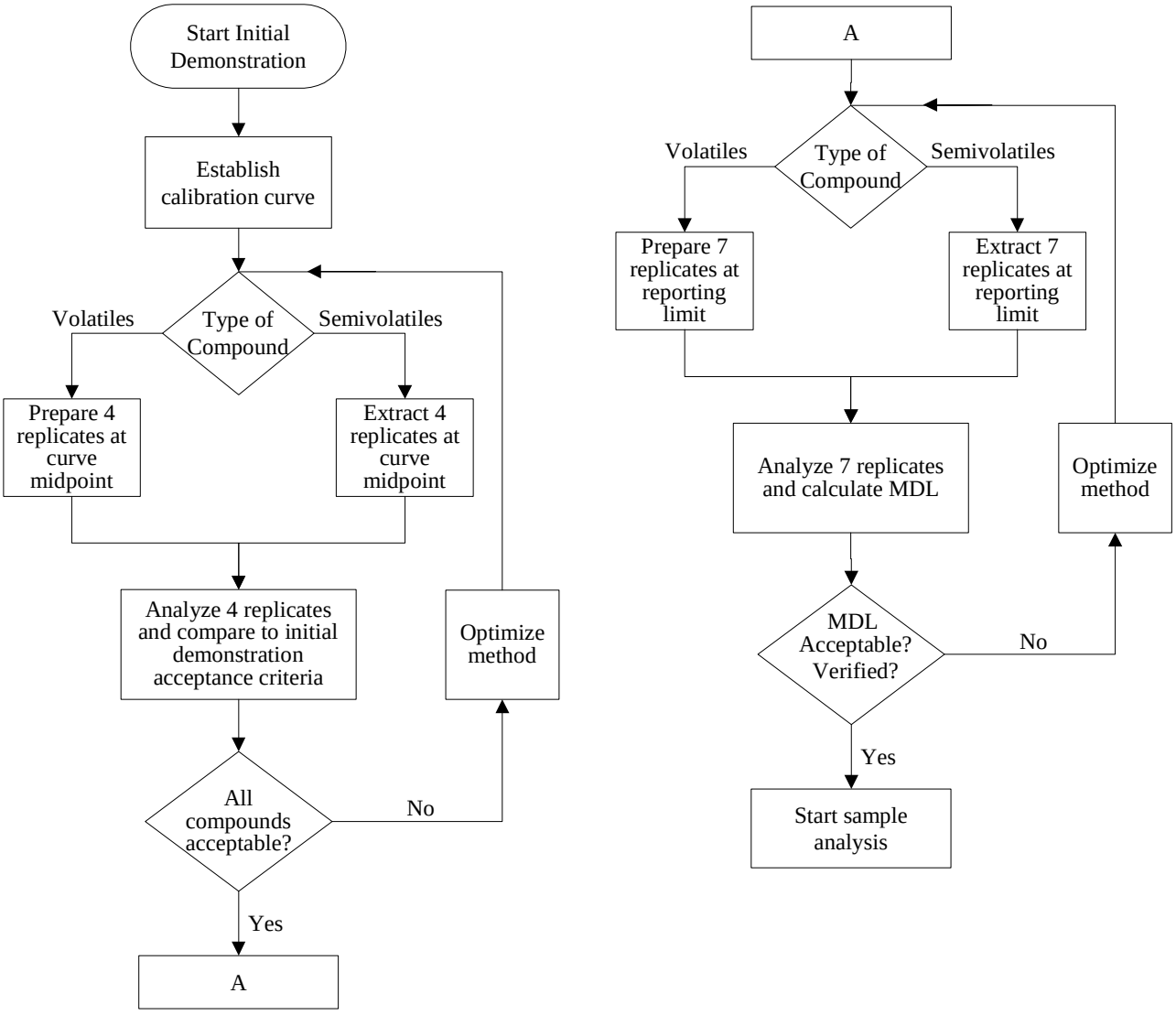

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Figure 2 - Sample Analysis[1] Flow Diagram

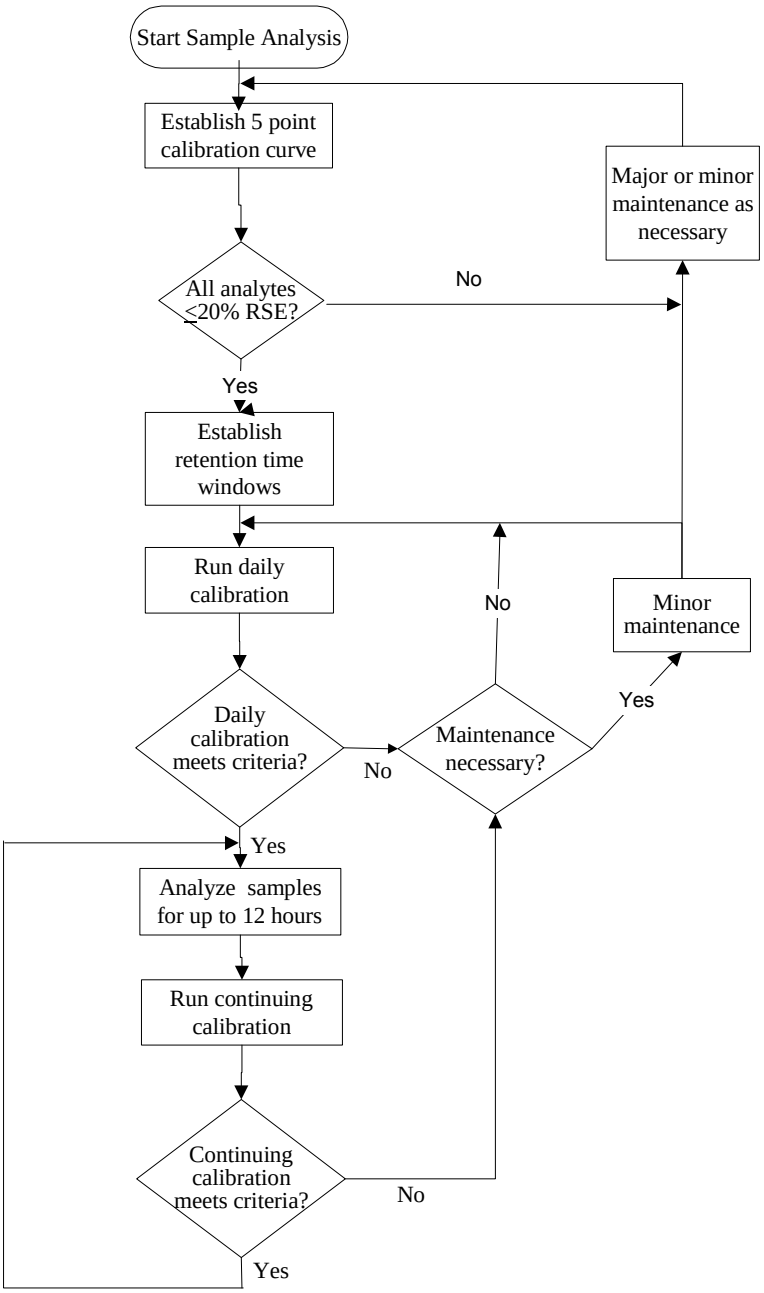

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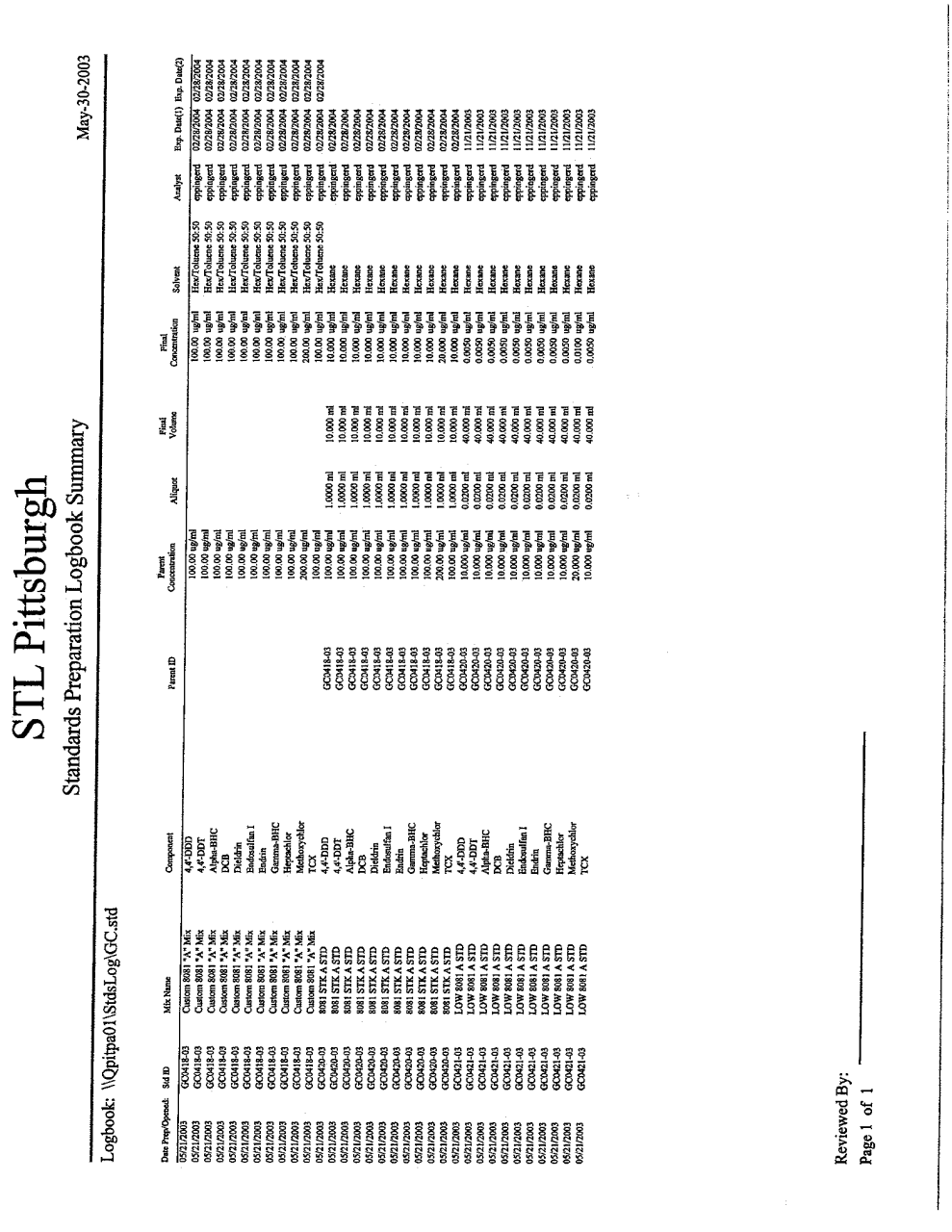
Figure 3 – Example Standard Preparation Logbook Page



STL Pittsburgh

Standards Preparation Logbook Summary

May-30-2003

Logbook: \\Qpitpa01\StdsLog\GC.std

Date Prep/Opened	Std ID	Mix Name	Component	Parent ID	Parent Concentration	Aliquot	Final Volume	Final Concentration	Solvent	Analyst	Exp. Date(1)	Exp. Date(2)
05/21/2003	GC0418-03	Custom 8081 "A" Mix	4,4'-DDD		100.00 ug/ml			100.00 ug/ml	Hex/Toluene 50:50	epplegard	02/28/2004	02/28/2004
05/21/2003	GC0418-03	Custom 8081 "A" Mix	4,4'-DDT		100.00 ug/ml			100.00 ug/ml	Hex/Toluene 50:50	epplegard	02/28/2004	02/28/2004
05/21/2003	GC0418-03	Custom 8081 "A" Mix	Alpha-BHC		100.00 ug/ml			100.00 ug/ml	Hex/Toluene 50:50	epplegard	02/28/2004	02/28/2004
05/21/2003	GC0418-03	Custom 8081 "A" Mix	DCB		100.00 ug/ml			100.00 ug/ml	Hex/Toluene 50:50	epplegard	02/28/2004	02/28/2004
05/21/2003	GC0418-03	Custom 8081 "A" Mix	Dieldrin		100.00 ug/ml			100.00 ug/ml	Hex/Toluene 50:50	epplegard	02/28/2004	02/28/2004
05/21/2003	GC0418-03	Custom 8081 "A" Mix	Endosulfan I		100.00 ug/ml			100.00 ug/ml	Hex/Toluene 50:50	epplegard	02/28/2004	02/28/2004
05/21/2003	GC0418-03	Custom 8081 "A" Mix	Endrin		100.00 ug/ml			100.00 ug/ml	Hex/Toluene 50:50	epplegard	02/28/2004	02/28/2004
05/21/2003	GC0418-03	Custom 8081 "A" Mix	Gamma-BHC		100.00 ug/ml			100.00 ug/ml	Hex/Toluene 50:50	epplegard	02/28/2004	02/28/2004
05/21/2003	GC0418-03	Custom 8081 "A" Mix	Heptachlor		100.00 ug/ml			100.00 ug/ml	Hex/Toluene 50:50	epplegard	02/28/2004	02/28/2004
05/21/2003	GC0418-03	Custom 8081 "A" Mix	Methoxychlor		100.00 ug/ml			200.00 ug/ml	Hex/Toluene 50:50	epplegard	02/28/2004	02/28/2004
05/21/2003	GC0418-03	Custom 8081 "A" Mix	TCX		100.00 ug/ml			100.00 ug/ml	Hex/Toluene 50:50	epplegard	02/28/2004	02/28/2004
05/21/2003	GC0420-03	8081 STK A STD	4,4-DDD	GC0418-03	100.00 ug/ml	1.0000 ml	10.000 ml	10.000 ug/ml	Hexane	epplegard	02/28/2004	
05/21/2003	GC0420-03	8081 STK A STD	4,4-DDT	GC0418-03	100.00 ug/ml	1.0000 ml	10.000 ml	10.000 ug/ml	Hexane	epplegard	02/28/2004	
05/21/2003	GC0420-03	8081 STK A STD	Alpha-BHC	GC0418-03	100.00 ug/ml	1.0000 ml	10.000 ml	10.000 ug/ml	Hexane	epplegard	02/28/2004	
05/21/2003	GC0420-03	8081 STK A STD	DCB	GC0418-03	100.00 ug/ml	1.0000 ml	10.000 ml	10.000 ug/ml	Hexane	epplegard	02/28/2004	
05/21/2003	GC0420-03	8081 STK A STD	Dieldrin	GC0418-03	100.00 ug/ml	1.0000 ml	10.000 ml	10.000 ug/ml	Hexane	epplegard	02/28/2004	
05/21/2003	GC0420-03	8081 STK A STD	Endosulfan I	GC0418-03	100.00 ug/ml	1.0000 ml	10.000 ml	10.000 ug/ml	Hexane	epplegard	02/28/2004	
05/21/2003	GC0420-03	8081 STK A STD	Endrin	GC0418-03	100.00 ug/ml	1.0000 ml	10.000 ml	10.000 ug/ml	Hexane	epplegard	02/28/2004	
05/21/2003	GC0420-03	8081 STK A STD	Gamma-BHC	GC0418-03	100.00 ug/ml	1.0000 ml	10.000 ml	10.000 ug/ml	Hexane	epplegard	02/28/2004	
05/21/2003	GC0420-03	8081 STK A STD	Heptachlor	GC0418-03	100.00 ug/ml	1.0000 ml	10.000 ml	10.000 ug/ml	Hexane	epplegard	02/28/2004	
05/21/2003	GC0420-03	8081 STK A STD	Methoxychlor	GC0418-03	200.00 ug/ml	1.0000 ml	10.000 ml	20.000 ug/ml	Hexane	epplegard	02/28/2004	
05/21/2003	GC0420-03	8081 STK A STD	TCX	GC0418-03	100.00 ug/ml	1.0000 ml	10.000 ml	10.000 ug/ml	Hexane	epplegard	02/28/2004	
05/21/2003	GC0421-03	LOW 8081 A STD	4,4'-DDD	GC0420-03	10.000 ug/ml	0.2000 ml	40.000 ml	0.0050 ug/ml	Hexane	epplegard	11/21/2003	
05/21/2003	GC0421-03	LOW 8081 A STD	4,4-DDT	GC0420-03	10.000 ug/ml	0.2000 ml	40.000 ml	0.0050 ug/ml	Hexane	epplegard	11/21/2003	
05/21/2003	GC0421-03	LOW 8081 A STD	Alpha-BHC	GC0420-03	10.000 ug/ml	0.2000 ml	40.000 ml	0.0050 ug/ml	Hexane	epplegard	11/21/2003	
05/21/2003	GC0421-03	LOW 8081 A STD	DCB	GC0420-03	10.000 ug/ml	0.2000 ml	40.000 ml	0.0050 ug/ml	Hexane	epplegard	11/21/2003	
05/21/2003	GC0421-03	LOW 8081 A STD	Dieldrin	GC0420-03	10.000 ug/ml	0.2000 ml	40.000 ml	0.0050 ug/ml	Hexane	epplegard	11/21/2003	
05/21/2003	GC0421-03	LOW 8081 A STD	Endosulfan I	GC0420-03	10.000 ug/ml	0.2000 ml	40.000 ml	0.0050 ug/ml	Hexane	epplegard	11/21/2003	
05/21/2003	GC0421-03	LOW 8081 A STD	Endrin	GC0420-03	10.000 ug/ml	0.2000 ml	40.000 ml	0.0050 ug/ml	Hexane	epplegard	11/21/2003	
05/21/2003	GC0421-03	LOW 8081 A STD	Gamma-BHC	GC0420-03	10.000 ug/ml	0.2000 ml	40.000 ml	0.0050 ug/ml	Hexane	epplegard	11/21/2003	
05/21/2003	GC0421-03	LOW 8081 A STD	Heptachlor	GC0420-03	10.000 ug/ml	0.2000 ml	40.000 ml	0.0050 ug/ml	Hexane	epplegard	11/21/2003	
05/21/2003	GC0421-03	LOW 8081 A STD	Methoxychlor	GC0420-03	20.000 ug/ml	0.2000 ml	40.000 ml	0.0100 ug/ml	Hexane	epplegard	11/21/2003	
05/21/2003	GC0421-03	LOW 8081 A STD	TCX	GC0420-03	10.000 ug/ml	0.2000 ml	40.000 ml	0.0050 ug/ml	Hexane	epplegard	11/21/2003	

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Appendix A

Analysis of PCB Congeners Based on Method 8082

19 SCOPE AND APPLICATION

19.1 This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polychlorinated biphenyls (PCB) congeners by GC/ECD. This Appendix is to be applied when SW-846 Method 8082 is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate TestAmerica sample extraction SOP (PITT-OP-0001). The PCBs are determined and quantitated as individual PCB congeners.

Table A-1 lists the congeners which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

20 SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts for PCB congeners. The PCBs are injected onto the GC column(s) and separated and detected by electron capture detection. Quantitation is by the external standard method.

21 DEFINITIONS

Refer to the LQM for definitions of terms used in this document.

22 INTERFERENCES

22.1 Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

22.2 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

22.3 Sulfur will interfere and can be removed using procedures described in SOP PITT-OP-0001.

22.4 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), Sulfur cleanup (Method 3660), and Acid Cleanup (Method 3665). These cleanup procedures are included in SOP PITT-OP-0001.

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23 SAFETY

23.1 Refer to section 5 of the Method 8000B SOP for general safety requirements.

23.2 PCB congeners have been classified as a potential carcinogen under OSHA. Concentrated solutions of PCB congeners must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

23.3 All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

23.4 All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

24 EQUIPMENT AND SUPPLIES

24.1 Refer to Section 6 of the 8000B section of this SOP. A GC equipped with a ^{63}Ni electron capture detector is required.

24.2 Refer to Table A-2 for analytical columns.

24.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

25 REAGENTS AND STANDARDS

25.1 Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

25.2 Refer to Tables A-3 and A-6 for details of calibration standards.

25.3 Surrogate Standards

Tetrachloro-m-xylene (TCMX) and the congener BZ-165 are the surrogate standards. Refer to Table A-4 for details of surrogate standards.

26 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

27 QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

28 PROCEDURE

28.1 Calibration and Standardization

28.2 Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

28.3 Initial Calibration

28.3.1.1 Refer to Table A-5 for the initial calibration analytical sequence.

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28.3.1.2 The response for each PCB congener will be calculated by the procedures described in the general method for GC analysis, with the following modifications.

28.3.1.2.1 A five-point calibration of each of the individual congeners mixes is generated. At least 2 separate mixes are prepared to ensure that there is complete resolution of all congeners in the mixes.

28.4 Initial Calibration Verification

28.4.1.1 The ICV will consist of second source standards of all congeners of interest. Refer to the 8000B section of this SOP for acceptance criteria for the Initial Calibration Verification.

28.5 12 hour Calibration

28.5.1.1 The 12-hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration.

28.5.1.2 The retention time windows for any analytes included in the daily calibration are updated.

28.5.1.3 For this method, samples must be bracketed with successful calibration verification runs.

28.6 Calibration Verification

28.6.1.1 A mid-level calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples or 12 hours, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.).

28.6.1.2 The daily CCV analysis at a concentration other than the mid level (to meet NELAC requirements) will consist of all of the congeners of interest. All other CCVs will be mid level calibration standards.

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Calibration Controls	Sequence	Control Limit
Initial Calibration Standards	5 pt. Curve (see Table A-3) prior to samples	≤20% RSD (alternatively, if the correlation coefficient is >0.99, linear regression may be used).
Second Source Verification	After initial calibration	± 20% Difference of expected value
Cont. Calib. Verif. (CCV)	After initial calibration, Every 20 samples	± 15% Difference*
Retention Time Windows	After initial calibration, update daily	3 X Std. Deviation
* For Non-Routine compounds (Table A-1) the CCV may be <25% unless otherwise specified by the project. **Note:** If the CCV is > ±15% for any one compound, data will be acceptable if the result is J flagged. The result must be less than the RL.		

28.7 Procedure

28.8 Refer to the method 8000B section of this SOP for general procedural requirements.

28.9 If one surrogate is out of control in a sample and all surrogates are in control for the method blank and LCS, then matrix effect has been demonstrated for the sample and repreparation is not necessary. The client may be contacted for input if the re-extraction is expected to take place after the sample holding time has been exceeded.

28.10 Extraction

The extraction procedure is described in SOP No. PITT-OP-0001.

28.11 Cleanup

Cleanup procedures are described in SOP No. PITT-OP-0001.

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28.12 Suggested gas chromatographic conditions are given in Table A-2.

28.13 Allow extracts to warm to ambient temperature before injection.

28.14 The suggested analytical sequence is given in Table A-5.

29 CALCULATIONS / DATA REDUCTION

29.1 Identification of Congeners

29.2 Retention time windows are used for identification of PCB congeners. Second column confirmation must be performed.

29.2.1.1 A few of the congeners listed in this SOP co elute on one of the GC columns but not on the other column. If those specific congeners are compounds of concern for a project, the GC column with no co elution will be the primary quantitation column.

29.3 Surrogate recovery results are calculated and reported for TCMX and BZ-205. Corrective action is only necessary if BZ-205 and TCMX are both outside of acceptance limits.

30 METHOD PERFORMANCE

30.1 Performance limits for the four replicate initial demonstration of capability required under Section 12.1 of the main body of this SOP are recoveries of 70-130% for all congeners listed in Table A-4. The spiking level should be equivalent to a mid level calibration.

30.2 Method detection limits (MDL) are determined for all congeners.

31 POLLUTION CONTROL

31.1 Refer to Section 13 of the 8000B section of this SOP.

32 WASTE MANAGEMENT

32.1 Refer to Section 14 of the 8000B section of this SOP.

33 REFERENCES

33.1 Test Methods for Evaluating Solid Waste, SW-846, Update III, December 1996, Method 8082

33.2 Ballschmiter, K. and M. Zell. 1980. Analysis of polychlorinated biphenyls (PCBs) by glass capillary gas chromatography, composition of technical aroclor- and clophen-PCB mixtures. Fresenius Anal. Chem. 302:20-31.

33.3 SOP # PITT-QA-DoD-0001, Implementation of DoD QSM Version 3 January 2006, current version.

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34 ATTACHMENTS

34.1 Table A-1: Standard Analyte List and Reporting Limits

34.2 Table A-2: Recommended GC Operating Conditions

34.3 Table A-3: Calibration Levels

34.4 Table A-4: LCS/Matrix Spike and Surrogate Spike Levels

34.5 Table A-5: Suggested Analytical Sequence

34.6 Table A-6: Preparation of Calibration Standards

34.7 Table A-7: Surrogate Recovery Limits

35 REVISION HISTORY

35.1 Revision 12, 09/07/2007

35.2 Changed the format of the SOP to correspond to the new Corporate SOP format.

36 METHOD MODIFICATIONS

36.1 Refer to Section 18 of the 8000B section of this SOP.

Table A-1

Standard Analyte List and Reporting Limits

Compound *	CAS #	Reporting Limit, ng/L or µg/kg				
		Water	Low level soil	High level soil	Tissue	Waste
BZ-1[1]	2051-60-7	10	1.7	8.50	--	51.0
BZ-3	2051-62-9	10	1.7	8.50	--	51.0
BZ-5[1]	16605-91-7	1.0	0.17	0.85	--	5.1
BZ-8	34883-43-7	1.0	0.17	0.85	2.0	5.1
BZ-15	2050-68-2	10	1.7	8.50	--	51.0
BZ-18[1]	37680-65-2	1.0	0.17	0.85	2.0	5.1
BZ-28	7012-37-5	1.0	0.17	0.85	2.0	5.1
BZ-31[1]	16606-02-3	1.0	0.17	0.85	--	5.1
BZ-37	38444-90-5	1.0	0.17	0.85	--	5.1
BZ-44[1]	41464-39-5	1.0	0.17	0.85	2.0	5.1
BZ-49	41464-40-8	1.0	0.17	0.85	2.0	5.1

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Table A-1

Standard Analyte List and Reporting Limits

Compound *	CAS #	Reporting Limit, ng/L or µg/kg				
		Water	Low level soil	High level soil	Tissue	Waste
BZ-52[1]	35693-99-3	1.0	0.17	0.85	2.0	5.1
BZ-66[1]	32598-10-0	1.0	0.17	0.85	2.0	5.1
BZ-70	32598-11-1	1.0	0.17	0.85	--	5.1
BZ-74	32690-93-0	1.0	0.17	0.85	--	5.1
BZ-77	32598-13-3	1.0	0.17	0.85	2.0	5.1
BZ-81	70362-50-4	1.0	0.17	0.85	--	5.1
BZ-87[1]	38380-02-8	1.0	0.17	0.85	2.0	5.1
BZ-90	68194-07-0	1.0	0.17	0.85	--	5.1
BZ-99	38380-01-7	1.0	0.17	0.85	--	5.1
BZ-101[1]	37680-73-2	1.0	0.17	0.85	2.0	5.1
BZ-105	32598-14-4	1.0	0.17	0.85	2.0	5.1
BZ-110[1]	38380-03-9	1.0	0.17	0.85	--	5.1
BZ-114	74472-37-0	1.0	0.17	0.85	--	5.1
BZ-115	74472-38-1	1.0	0.17	0.85	--	5.1
BZ-118	31508-00-6	1.0	0.17	0.85	2.0	5.1
BZ-119	56558-17-9	1.0	0.17	0.85	--	5.1
BZ-123	65510-44-3	1.0	0.17	0.85	--	5.1
BZ-126	57465-28-8	1.0	0.17	0.85	2.0	5.1
BZ-128	38380-07-3	1.0	0.17	0.85	2.0	5.1
BZ-138[1]	35065-28-2	1.0	0.17	0.85	2.0	5.1
BZ-141[1]	52712-04-6	1.0	0.17	0.85	--	5.1
BZ-149	38380-04-0	1.0	0.17	0.85	--	5.1
BZ-151[1]	52663-63-5	1.0	0.17	0.85	--	5.1
BZ-153[1]	35065-27-1	1.0	0.17	0.85	2.0	5.1

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Table A-1

Standard Analyte List and Reporting Limits

Compound *	CAS #	Reporting Limit, ng/L or µg/kg				
		Water	Low level soil	High level soil	Tissue	Waste
BZ-156	38380-08-4	1.0	0.17	0.85	2.0	5.1
BZ-157	69782-90-7	1.0	0.17	0.85	--	5.1
BZ-158	74472-42-7	1.0	0.17	0.85	--	5.1
BZ-167	52663-72-6	1.0	0.17	0.85	--	5.1
BZ-168	59291-65-5	1.0	0.17	0.85	--	5.1
BZ-169	32774-16-6	1.0	0.17	0.85	2.0	5.1
BZ-170[1]	35065-30-6	1.0	0.17	0.85	2.0	5.1
BZ-177	52663-70-4	1.0	0.17	0.85	--	5.1
BZ-180[1]	35065-29-3	1.0	0.17	0.85	2.0	5.1
BZ-183[1]	52663-69-1	1.0	0.17	0.85	2.0	5.1
BZ-184	74472-48-3	1.0	0.17	0.85	2.0	5.1
BZ-187[1]	52663-68-0	1.0	0.17	0.85	2.0	5.1
BZ-189	39635-31-9	1.0	0.17	0.85	--	5.1
BZ-194	35694-08-7	1.0	0.17	0.85	--	5.1
BZ-195	52663-78-2	1.0	0.17	0.85	2.0	5.1
BZ-199	52663-73-7	1.0	0.17	0.85	--	5.1
BZ-200	40186-71-8	1.0	0.17	0.85	--	5.1
BZ-201	52663-75-9	1.0	0.17	0.85	--	5.1
BZ-202	2136-99-4	1.0	0.17	0.85	--	5.1
BZ-206[1]	40186-72-9	1.0	0.17	0.85	2.0	5.1
BZ-207	52663-79-3	1.0	0.17	0.85	--	5.1
BZ-209	2051-24-3	1.0	0.17	0.85	2.0	5.1

[1] The congeners footnoted are routinely analyzed. All others are considered non-routine.

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* The congener identifications are consistent with the short-hand identifications recommended by Ballschmiter and Zell (1980).

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.	Dilution Factor
Groundwater	1000 mL	2 mL	1
Low-Level Soil	12 g	4 mL	1
High-Level Soil	12 g	4 mL	5
Waste	1 g	10 mL	1
Tissue	5 g	10 mL (5 mL with GPC clean-up)	

Table A-2

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	310
Temperature program	100C for 1.0min, 4°C/min to 292
Column 1	ZB 50, 30 m, 0.25 mm id, 0.25 µm FT
Column 2	ZB 1701, 30 m, 0.25 mm id, 0.25 µm FT
Injection	1-2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen

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Table A-3					
Calibration Levels ug/mL					
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**
BZ-1	**0.005**	**0.01**	**0.025**	**0.050**	**0.10**
BZ-3	0.005	0.01	0.025	0.050	0.10
BZ-5	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-18	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-15	0.005	0.01	0.025	0.050	0.10
BZ-28	0.0005	0.001	0.0025	0.0050	0.010
BZ-31	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-37	0.0005	0.001	0.0025	0.0050	0.010
BZ-44	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-49	0.0005	0.001	0.0025	0.0050	0.010
BZ-52	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-66	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-70	0.0005	0.001	0.0025	0.0050	0.010
BZ-74	0.0005	0.001	0.0025	0.0050	0.010
BZ-77	0.0005	0.001	0.0025	0.0050	0.010
BZ-81	0.0005	0.001	0.0025	0.0050	0.010
BZ-87	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-90	0.0005	0.001	0.0025	0.0050	0.010
BZ-99	0.0005	0.001	0.0025	0.0050	0.010
BZ-101	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-105	0.0005	0.001	0.0025	0.0050	0.010
BZ-110	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-114	0.0005	0.001	0.0025	0.0050	0.010
BZ-115	0.0005	0.001	0.0025	0.0050	0.010

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Table A-3					
Calibration Levels ug/mL					
BZ-118	0.0005	0.001	0.0025	0.0050	0.010
BZ-119	0.0005	0.001	0.0025	0.0050	0.010
BZ-123	0.0005	0.001	0.0025	0.0050	0.010
BZ-126	0.0005	0.001	0.0025	0.0050	0.010
BZ-128	0.0005	0.001	0.0025	0.0050	0.010
BZ-138	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-141	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-149	0.0005	0.001	0.0025	0.0050	0.010
BZ-151	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-153	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-156	0.0005	0.001	0.0025	0.0050	0.010
BZ-157	0.0005	0.001	0.0025	0.0050	0.010
BZ-158	0.0005	0.001	0.0025	0.0050	0.010
BZ-167	0.0005	0.001	0.0025	0.0050	0.010
BZ-168	0.0005	0.001	0.0025	0.0050	0.010
BZ-169	0.0005	0.001	0.0025	0.0050	0.010
BZ-170	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-177	0.0005	0.001	0.0025	0.0050	0.010
BZ-180	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-183	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-184	0.0005	0.001	0.0025	0.0050	0.010
BZ-187	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-189	0.0005	0.001	0.0025	0.0050	0.010
BZ-194	0.0005	0.001	0.0025	0.0050	0.010
BZ-195	0.0005	0.001	0.0025	0.0050	0.010

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Table A-3					
Calibration Levels ug/mL					
BZ-200	0.0005	0.001	0.0025	0.0050	0.010
BZ-201	0.0005	0.001	0.0025	0.0050	0.010
BZ-202	0.0005	0.001	0.0025	0.0050	0.010
BZ-206	**0.0005**	**0.001**	**0.0025**	**0.0050**	**0.010**
BZ-207	0.0005	0.001	0.0025	0.0050	0.010
BZ-209	0.0005	0.001	0.0025	0.0050	0.010
Note: The PCB Congeners in bold are the routine ones.					
SURROGATES					
TCMX	0.00083	0.00166	0.00416	0.00833	0.01666
BZ-205	0.00083	0.00166	0.00416	0.00833	0.01666

Table A-4					
LCS/Matrix Spike and Surrogate Spike levels for Congener Analysis					
ng/L or µg/kg					
Compound	**Aqueous**	**Low Level Soil**	**High Level Soil**	**Tissue**	**Waste**
BZ-8	10	1.67	8.33	10	50
BZ-18	10	1.67	8.33	10	50
BZ-28	10	1.67	8.33	10	50
BZ-44	10	1.67	8.33	10	50
BZ-49	10	1.67	8.33	10	50
BZ-52	10	1.67	8.33	10	50
BZ-66	10	1.67	8.33	10	50
BZ-77	10	1.67	8.33	10	50
BZ-87	10	1.67	8.33	10	50
BZ-101	10	1.67	8.33	10	50

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Table A-4

LCS/Matrix Spike and Surrogate Spike levels for Congener Analysis

ng/L or µg/kg

BZ-105	10	1.67	8.33	10	50
BZ-118	10	1.67	8.33	10	50
BZ-126	10	1.67	8.33	10	50
BZ-128	10	1.67	8.33	10	50
BZ-138	10	1.67	8.33	10	50
BZ-153	10	1.67	8.33	10	50
BZ-156	10	1.67	8.33	10	50
BZ-169	10	1.67	8.33	10	50
BZ-170	10	1.67	8.33	10	50
BZ-180	10	1.67	8.33	10	50
BZ-183	10	1.67	8.33	10	50
BZ-184	10	1.67	8.33	10	50
BZ-187	10	1.67	8.33	10	50
BZ-195	10	1.67	8.33	10	50
BZ-206	10	1.67	8.33	10	50
BZ-209	10	1.67	8.33	10	50
Surrogates					
TCMX	5	0.8333	13.33	16	80
BZ-205	5	0.8333	13.33	16	80

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Table A-5

Suggested Analytical Sequence

Initial Calibration		
Injection #		
Solvent blank (optional)		
PCB Congener Mix 1	5-point	5-point
PCB Congener Mix 2	5-point	5-point
ICV (second source standard(s) of all congeners of interest)		
Samples 1-20 (or 12 hours)		
Solvent blank (optional)		
PCB Congener Mix 1	Level 3	Level 3
PCB Congener Mix 2	Level 3	Level 3, etc.
After 12 hours:		
PCB Congener Mix 1	Level 3	Level 3
PCB Congener Mix 2	Level 3	Level 3
Samples 1-20 (or 12 hours)		
Solvent blank (optional)		
PCB Congener Mix 1	Level 3	Level 3
PCB Congener Mix 2	Level 3	Level 3, etc.
12 hour Calibration		

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated.

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Table A-6

Preparation of Calibration Standards

Calibration Level	Mix 1 Intermediate (uL)	Mix 2 Intermediate (uL)
Level 1	50	50
Level 2	100	100
Level 3	625	625
Level 4	500	500
Level 5	1000	1000

The congener stock standards are purchased as certified standards in two separate solutions in isooctane. The Mix 1 stock includes BZ# 8, 18, 28, 44, 49, 52, 66, 77, 87, 101, 105, 118, 126, 128, 138, 153, 156, 169, 170, 180, 183, 184, 187, 195, 206, and 209 at 4.0 ug/mL, and BZ# 165 (surrogate), and Tetrachloro-m-xylene (surrogate) at 6.66 ug/mL. The Mix 2 stock includes BZ# 1, 3, and 15 at 40 ug/mL, and BZ# 5, 31, 37, 70, 74, 81, 90, 99, 110, 114, 115, 119, 123, 141, 149, 151, 157, 158, 167, 168, 177, 189, 194, 201, 202, and 207 at 4.0 ug/mL. An intermediate Mix 1 and Mix 2 standard is prepared by diluting 1.0 mL of each of the appropriate stock mix to 10.0 mL in hexane. The intermediate Mix 1 concentrations are 0.40 ug/mL for each congener and 0.666ug/mL for each surrogate. The intermediate Mix 2 concentrations are 0.40 ug/mL for each congener except BZ# 1, 3 and 15, which are at 4.0 ug/mL. The working standards are prepared by diluting the volume noted in Table A-6 to a 40.0 mL final volume in hexane except for the Level 3 standard, which is taken to a 100 mL final volume in hexane.

Table A-7

Surrogate Recovery Limits

Surrogate	Water Recovery Limits	Soil Recovery Limits
PCB 205(BZ)	30-130	30-130
Tetrachloro-m-xylene	30-150	35-150

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Appendix B

Analysis of Organochlorine Pesticides Based on Method 8081A

37 SCOPE AND APPLICATION

This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of organochlorine pesticides by GC/ECD. This Appendix is to be applied when SW-846 Method 8081A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate TestAmerica sample extraction SOP (PITT-OP-0001)

Table B-1 lists compounds that are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

At client request, this method may also be used for the analysis of PCBs (Aroclors) in combination with pesticides, although these are normally analyzed following method 8082, as described in Appendix C of this SOP. In any event, if samples for PCB analysis do not need the acid clean up procedure, then the same injection may be used for method 8081A and 8082, assuming all calibration and QC requirements for both methods are met. Extracts that have been acid cleaned may not be analyzed for pesticides, since several of the pesticides will be degraded.

38 SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts for organochlorine pesticides. The pesticides are injected onto the GC column(s) and separated and detected by electron capture detection. Quantitation is by the external standard methods.

39 DEFINITIONS

Refer to the LQM for definitions of terms used in this document.

40 INTERFERENCES

40.1 Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

40.2 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

40.3 Sulfur will interfere and can be removed using procedures described in SOP PITT-OP-0001.

40.4 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and

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Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # PITT-OP-0001. Use of hexane / acetone as the extraction solvent (rather than acetone / methylene chloride) may reduce the amount of interferences extracted.

41 SAFETY

41.1 Refer to Section 5 of the Method 8000B SOP for general safety requirements.

41.2 Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

41.3 All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

41.4 All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

42 EQUIPMENT AND SUPPLIES

42.1 Refer to Section 6 of the 8000B section of this SOP. A GC equipped with a ^{63}Ni electron capture detector is required.

42.2 Refer to Table B-2 for analytical columns.

42.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

43 REAGENTS AND STANDARDS

43.1 Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

43.2 Refer to Tables B-3 and B-9 for details of calibration standards.

43.3 Surrogate Standards

Tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) are the surrogate standards. Refer to Tables B-5 and B-6 for details of surrogate standards.

43.4 Column Degradation Evaluation Mix

A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after one year, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. This solution also contains the surrogates. Refer to Table B-4 for details of the column degradation evaluation mix.

44 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

45 QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

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46 PROCEDURE

46.1 Calibration and Standardization

46.2 Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

46.3 Refer to Table B-2 for details of GC operating conditions. The conditions listed should result in resolution of all analytes listed in Table B-1 on both columns.

46.4 Column Degradation Evaluation

The column evaluation mix must be injected before each initial or daily calibration. The degradation of DDT and endrin must be calculated (see Section 11.8) and each shown to be less than 15% before calibration can proceed. The entire calculation must be included with the raw data and is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.

If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:

Replacement of the injection port liner or the glass wool.

Cutting off a portion of the injection end of a capillary column.

Replacing the GC column.

46.5 Initial Calibration

46.5.1.1 Refer to Section 10 of the 8000B section of this SOP for details of calibration procedures.

46.5.1.2 Refer to Table B-7 for the initial calibration analytical sequence.

46.5.1.3 The response for each single-peak analyte will be calculated by the procedures described in the general method for GC analysis.

46.5.1.4 The surrogate calibration curve is calculated from the Pesticide Mix. Surrogates in the other calibration standards are used only as retention time markers.

46.5.1.5 For multi-component pesticides: Single point calibration is used for multicomponent pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to section 11.1.4 for guidance on which option to use.

46.5.1.6 For multicomponent analytes, the mid level standard must be analyzed as part of the initial calibration. This single point calibration is used to quantitate multicomponent analytes.

46.5.1.7 The analyst may include a full 5 point calibration for any of the multicomponent analytes with the initial calibration.

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46.6 Initial Calibration Verification

46.6.1.1 The ICV will consist of second source standards of all single peak analytes. Refer to the 8000B section of this SOP for acceptance criteria.

46.7 12 hour Calibration Verification

46.7.1.1 The 12 hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration. A mid level calibration standard is used for the 12 hour calibration. Refer to the 8000B section of this SOP for acceptance criteria.

46.7.1.2 At a minimum, the 12 hour calibration includes analysis of the breakdown mix followed by mid level standards of any single and multicomponent analytes.

46.7.1.3 The retention time windows for any analytes included in the 12 hour calibration are updated.

46.8 Continuing Calibration

46.9 The pesticide calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. If 12 hours elapse analyze the 12 hour standard sequence instead. The continuing calibration standard need not include multicomponent analytes. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

46.10 The daily CCV analysis at a concentration other than the mid level (to meet NELAC requirements) will consist of all of the single peak analytes. All other CCVs will be mid level calibration standards.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5 pt. Curve (minimum)	$\leq$ 20% RSD (alternatively, if the correlation coefficient is >0.99, linear regression may be used).
Second Source	prior to samples	$\pm$ 20 % of expected value
Retention Time Windows	after calibration, update daily	3 X Standard Deviation
Cont. Cal. Verif. (CCV)	every 20 samples	± 15% D*
* For Non-Routine compounds (Table B-1) the CCV may be < 25% unless otherwise specified by the project. **Note:** If the CCV is > ±15% for any one compound, data will be acceptable if the result is J flagged. The result must be less than the RL.		

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46.11 Procedure

46.12 Refer to the method 8000B section of this SOP for general procedural requirements.

46.13 Extraction

The extraction procedure is described in SOP No. PITT-OP-0001.

46.14 Cleanup

Cleanup procedures are described in SOP No. PITT-OP-0001.

46.15 An additional cleanup (Carboprep 90) may be performed prior to analysis if the sample extract has some color. Refer to Section 10.2.3.2 of this SOP for the Carboprep 90 Cleanup procedure.

46.16 Suggested gas chromatographic conditions are given in Table B-2.

46.17 Allow extracts to warm to ambient temperature before injection.

46.18 The suggested analytical sequence is given in Table B-7.

47 CALCULATIONS / DATA REDUCTION

47.1 Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

47.2 Identification of Multicomponent Analytes

Retention time windows are also used for identification of multi-component analytes, but the “fingerprint” produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst’s judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

47.3 Quantitation of Multicomponent Analytes

47.4 Use 3-5 major peaks or total area for quantitation as described in section 11.1.4, initial calibration of multicomponent analytes.

47.5 If there are no interfering peaks within the envelope of the multicomponent analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

47.5.1.1 Multiple peak option

This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 3-5 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-5 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in

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samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

Chlordane may be quantitated either using the multiple peak option total area option or by quantitation of the major components, α-chlordane, γ-chlordane and heptachlor.

47.5.1.2 Total area option

The total area of the standards and samples may be used for quantitation of multicomponent analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multicomponent pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

47.6 Second column confirmation for multi-component analytes will only be performed when requested by the client, because the appearance of the multi-peak "fingerprint" in the sample usually serves as a confirmation of analyte presence.

47.7 Surrogate recovery results are calculated and reported for tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) in all samples. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

47.8 Calculation of Column Degradation/% Breakdown (%B)

$$DDT\ \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

where:

A_{DDD}, A_{DDE}, and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

$$Endrin\ \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100$$

where:

A_{EK}, A_{EA}, and A_E = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

48 METHOD PERFORMANCE

48.1 Performance limits for the four replicate initial demonstration of capability required under Section 12.2 of the main body of this SOP are presented in Table B-8. The spiking level should be equivalent to a mid level calibration.

49 POLLUTION CONTROL

Refer to Section 13 of the 8000B section of this SOP.

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50 WASTE MANAGEMENT

50.1 Refer to Section 14 of the 8000B section of this SOP.

51 REFERENCES

51.1 Test Methods for Evaluating Solid Waste, SW-846, Update III, December 1996, Method 8081A

51.2 SOP # PITT-QA-DoD-0001, Implementation of DoD QSM Version 3 January 2006, current version.

52 ATTACHMENTS

52.1 Table B-1: Standard Analyte List and Reporting Limits

52.2 Table B-2: Recommended GC Operating Conditions

52.3 Table B-3: Calibration Levels

52.4 Table B-4: Column Degradation Evaluation Mix

52.5 Table B-5: LCS/Matrix Spike and Surrogate Spike Levels

52.6 Table B-6: LCS/Matrix Spike and Surrogate Spike Levels for TCLP

52.7 Table B-7: Suggested Analytical Sequence

52.8 Table B-8: Performance Limits for Four Replicate Initial Demonstration of Capability

52.9 Table B-9: Preparation of Calibration Standards

52.10 Table B-10: Surrogate Recovery Limits

53 REVISION HISTORY

53.1 Revision 12, 09/07/2007

53.2 Changed the format of the SOP to correspond to the new Corporate SOP format.

54 METHOD MODIFICATIONS

54.1 Refer to Section 18 of the 8000B section of this SOP.

Table B-1

Standard Analyte List and Reporting Limits (µg/L, µg/wipe or µg/kg)

Compound	CAS #	Water or Wipe/TCLP	Soil/Low-level Soil	Tissue	Waste	Lowlevel Water
Aldrin	309-00-2	0.05	1.7/0.0833	1.7	50	0.0013
α-BHC	319-84-6	0.05	1.7/0.0833	1.7	50	0.0013
β-BHC	319-85-7	0.05	1.7/0.0833	1.7	50	0.0013

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Table B-1

Standard Analyte List and Reporting Limits (µg/L, µg/wipe or µg/kg)

Compound	CAS #	Water or Wipe/TCLP	Soil/Low-level Soil	Tissue	Waste	Lowlevel Water
δ-BHC	319-86-8	0.05	1.7/0.0833	1.7	50	0.0013
γ-BHC (Lindane)	58-89-9	0.05/0.5	1.7/0.0833	1.7	50	0.0013
α-Chlordane	5103-71-9	0.05	1.7/0.0833	1.7	50	0.0013
γ-Chlordane	5103-74-2	0.05	1.7/0.0833	1.7	50	0.0013
Chlordane (tech.)	57-74-9	0.5/5.0	17/0.0833	17	500	0.0125
4,4'-DDD	72-54-8	0.05	1.7/0.0833	1.7	50	0.0013
4,4'-DDE	72-55-9	0.05	1.7/0.0833	1.7	50	0.0013
4,4'-DDT	50-29-3	0.05	1.7/0.0833	1.7	50	0.0013
Dieldrin	60-57-1	0.05	1.7/0.0833	1.7	50	0.0013
Endosulfan I	959-98-8	0.05	1.7/0.0833	1.7	50	0.0013
Endosulfan II	33213-65-9	0.05	1.7/0.0833	1.7	50	0.0013
Endosulfan Sulfate	1031-07-8	0.05	1.7/0.0833	1.7	50	0.0013
Endrin	72-20-8	0.05/0.5	1.7/0.0833	1.7	50	0.0013
Endrin Aldehyde	7421-93-4	0.05	1.7/0.0833	1.7	50	0.0013
Endrin ketone	53494-70-5	0.05	1.7/0.0833	1.7	50	0.0013
Heptachlor	76-44-8	0.05/0.5	1.7/0.0833	1.7	50	0.0013
Heptachlor Epoxide	1024-57-3	0.05/0.5	1.7/0.0833	1.7	50	0.0013
Methoxychlor	72-43-5	0.1/1.0	3.3/0.1666	3.3	100	0.0025
Toxaphene	8001-35-2	2.0/20	67/3.333	67	2000	0.0500
NON ROUTINE STANDARDS						
2,4'-DDE	3424-82-6	0.05	1.7	--	50	0.0013
2,4'-DDD	53-19-0	0.05	1.7	--	50	0.0013
Cis-Nonachlor	5103-73-1	--	1.7	--	--	0.0013
Trans-Nonachlor	39765-80-5	--	1.7	--	--	0.0013

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Table B-1

Standard Analyte List and Reporting Limits (µg/L, µg/wipe or µg/kg)

Compound	CAS #	Water or Wipe/TCLP	Soil/Low-level Soil	Tissue	Waste	Lowlevel Water
Hexachlorobutadiene	87-68-3	--	1.7	--	--	--
2,4'-DDT	789—02-6	0.05	1.7	--	50	0.0030
Chlorbenside	103-17-3	0.1	3.3	3.3	100	0.0032
Dacthal (DCPA)	1861-32-1	0.1	3.3	3.3	100	0.0025
Hexachlorobenzene	118-74-1	0.05	1.7	--	50	0.0013
Hexachlorocyclopentadiene	77-47-4	0.1	3.3	--	100	--
Mirex	2385-85-5	0.05	1.7	1.7	50	0.0013
Diallate	2303-16-4	1.0	33	--	990	0.025
Isodrin	465-73-6	0.05	1.7	--	50	0.0013
Chlorobenzillate	510-15-6	0.5	17	--	500	0.0373

The following concentration factors are assumed when calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Groundwater	1000 mL	40 mL (1 mL for low-level)
TCLP Leachate	100 mL	40 mL
Soil	15 g	20 mL (1 mL for low-level)
Wipe	1 wipe	40 mL
High-Level Solid Waste	1 g	40 mL
Tissue	5 g	1 mL (with GPC clean-up)

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Table B-2	
Parameter	**Recommended Conditions**
Injection port temp	220°C
Detector temp	325°C
Temperature program	120°C for 1 min, 8.5°C/min to 285°C, , 6 min hold
Column 1	MR1, 30m X 0.53 mm id, 0.5µm
Column 2	MR2, 30m X 0.53 mm id, 0.5µm
Injection	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table B-3						
Calibration Levels ng/mL						
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6[2]**
Individual Mix A and B[1]						
Aldrin	1	5	25	50	100	200
g-BHC (Lindane)	1	5	25	50	100	200
Heptachlor	1	5	25	50	100	200
Methoxychlor	2	10	50	100	200	400
Dieldrin	1	5	25	50	100	200
Endosulfan I	1	5	25	50	100	200
Endosulfan II	1	5	25	50	100	200
4,4'-DDT	1	5	25	50	100	200
Endrin Aldehyde	1	5	25	50	100	200
Endrin Ketone	1	5	25	50	100	200
β-BHC	1	5	25	50	100	200

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Table B-3						
Calibration Levels ng/mL						
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6[2]**
δ-BHC	1	5	25	50	100	200
α-BHC	1	5	25	50	100	200
4,4'-DDD	1	5	25	50	100	200
4,4'-DDE	1	5	25	50	100	200
Endosulfan Sulfate	1	5	25	50	100	200
Endrin	1	5	25	50	100	200
α-Chlordane [3]	1	5	25	50	100	200
γ-Chlordane [3]	1	5	25	50	100	200
Heptachlor Epoxide	1	5	25	50	100	200
Multicomponent Standards						
Chlordane (Technical)			250[4]			
Toxaphene			1000[5]			
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	1	5	25	50	100	200
Decachlorobiphenyl	1	5	25	50	100	200

[1] Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.
[2] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.
[3] Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.
[4] This standard may be used for quantitation of technical chlordane between 50 and 1000 ng/mL. If the chlordane is more concentrated, the extract must be diluted and reanalyzed.
[5] This standard may be used for quantitation of toxaphene between 200 and 4000 ng/mL. If the toxaphene is more concentrated, the extract must be diluted and reanalyzed.

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Table B-4	
Column Degradation Evaluation Mix ng/mL	
Component	**Concentration**
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table B-5		
LCS/Matrix Spike and Surrogate Spike levels µg/L, µg/wipe or µg/kg		
	Aqueous/Wipe	**Soil/Tissue**
Aldrin	0.25	8.33
g-BHC (Lindane)	0.25	8.33
Heptachlor	0.25	8.33
Methoxychlor	0.25	8.33
Dieldrin	0.25	8.33
Endosulfan I	0.25	8.33
Endosulfan II	0.25	8.33
4,4'-DDT	0.25	8.33
Endrin Aldehyde	0.25	8.33
Endrin Ketone	0.25	8.33
β-BHC	0.25	8.33
δ-BHC	0.25	8.33
α-BHC	0.25	8.33
4,4'-DDD	0.25	8.33
4,4'-DDE	0.25	8.33
Endosulfan Sulfate	0.25	8.33
Endrin	0.25	8.33

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Table B-5		
LCS/Matrix Spike and Surrogate Spike levels µg/L, µg/wipe or µg/kg		
	Aqueous/Wipe	**Soil/Tissue**
α-Chlordane [3]	0.25	8.33
γ-Chlordane [3]	0.25	8.33
Heptachlor Epoxide	0.25	8.33
Tetrachloro-m-xylene (Surrogate)	0.2	6.67
Decachlorobiphenyl (Surrogate)	0.2	6.67

Table B-6	
LCS/Matrix Spike and Surrogate Spike levels for TCLP (µg/L)	
Heptachlor	2.5
Heptachlor epoxide	2.5
Lindane	2.5
Endrin	2.5
Methoxychlor	2.5
Tetrachloro-m-xylene (Surrogate)	2
Decachlorobiphenyl (Surrogate)	2

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Table B-7	
Suggested Analytical Sequence	
Initial Calibration	
Solvent Blank (optional)	
Breakdown Mix	
Pesticide Mix	All Levels
Technical Chlordane	Level 3[1]
Toxaphene	Level 3[1]
ICV (all single component analytes)	
Solvent Blank (optional)	
Up to 20 samples (unless 12 hours comes first)	
Solvent Blank (optional)	
Pesticide Mix	Mid-Level (continuing calibration), etc.
After 12 hours:	
Solvent Blank (optional)	
Breakdown Mix	
Pesticide Mix	
Any Multi-component Analytes	
Solvent Blank (optional)	
Up to 20 samples (unless 12 hours comes first)	
Solvent Blank (optional	
Pesticide Mix	Mid-Level (continuing calibration), etc.

[1]A five point curve for any of the multicomponent analytes may be included If Aroclors are included, a 5 point calibration for Aroclor 1016/1260 should be included with the initial calibration and a single point for the other Aroclors. The mid point 1016/1260 mix is included with the daily calibration (every 12 hours).

12-Hour Calibration
At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Pesticide Mix, and the breakdown mix must be run before the continuing calibration.

Table B-8

Performance limits, four replicate initial demonstration of capability

Compound	Initial demonstration, mean recovery limits	Initial demonstration, RSD limits
Aldrin	46-112	21
alpha-BHC	51-122	24
beta-BHC	61-120	32
delta-BHC	49.5-118.5	36
Gamma-BHC	57-116	23
Chlordane	44.8-108.6	20
4,4'-DDD	52-126	28
4,4'-DDE	46-120	27.5
4,4'-DDT	54-137	36
Dieldrin	42.5-124.5	38
Endosulfan I	43-141	24.5
Endosulfan II	78-171	61
Endosulfan Sulfate	62-132	27
Endrin	49-126	37
Heptachlor	57-100	20
Heptachlor Epoxide	43.5-131.5	25.4
Toxaphene	44.4-111.2	20

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Table B-9			
Preparation of Calibration Standards			
Calibration Level	**Intermediate (uL)**	**Toxaphene Stock (uL)**	**Chlordane Stock (uL)**
Level 1	4		
Level 2	20		
Level 3	250	400	100
Level 4	200		
Level 5	400		
Level 6	800		

The single peak pesticide stock standards are purchased as certified standards in two separate solutions in 50%hexane/50% toluene, which are combined prior to standard preparation. The A Mix stock includes alpha-BHC, Dieldrin, Endosulfan I, Endrin, gamma-BHC, Heptachlor, 4,4'-DDD, 4,4'-DDT, Decachlorobiphenyl (surrogate) and Tetrachloro-m-xylene (surrogate) at 100 ug/mL; and Methoxychlor at 200 ug/mL. The B Mix stock includes Aldrin, alpha-Chlordane, beta-BHC, delta-BHC, Endosulfan II, Endosulfan sulfate, Endrin aldehyde, Endrin ketone, gamma-Chlordane, Heptachlor epoxide, and 4,4'-DDE at 100 ug/mL. An intermediate standard is prepared by diluting 1.0 mL of each of the appropriate stock mix to 10.0 mL in hexane. The intermediate mix concentrations are 10 ug/mL for all compounds except methoxychlor, which is 20 ug/mL. The working standards are prepared by diluting the volume noted in Table B-9 to a 40.0 mL final volume in hexane except for the Level 3 standard, which is taken to a 100 mL final volume in hexane.

Toxaphene and Technical Chlordane stock standards are purchased certified solutions at 100 ug/mL. The mid level (Level 3) Toxaphene calibration standard is prepared by diluting 0.40 mL of the stock standard mix to 40 mL in hexane. The mid level (Level 3) Technical Chlordane calibration standard is prepared by diluting 0.10 mL of the stock standard mix to 40 mL in hexane.

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Table B-10		
Surrogate Recovery Limits		
Surrogate	**Water Recovery Limits**	**Soil Recovery Limits**
Decachlorobiphenyl	10-147	18-145
Tetrachloro-m-xylene	39-130	31-131

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Appendix C

Analysis of PCB Aroclors Based on Method 8082

55 SCOPE AND APPLICATION

55.1 This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polychlorinated biphenyls (PCBs) as Aroclors by GC/ECD. This Appendix is to be applied when SW-846 Method 8082 is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate TestAmerica sample extraction SOP (PITT-OP-0001). The PCBs are determined and quantitated as Aroclors.

Table C-1 lists the Aroclors, which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

56 SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts for PCB Aroclors. The PCBs are injected onto the GC column and separated and detected by electron capture detection. Quantitation is by the external standard method.

57 DEFINITIONS

Refer to the LQM for definitions of terms used in this document.

58 INTERFERENCES

58.1 Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

58.2 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

58.3 Sulfur will interfere and can be removed using procedures described in SOP PITT-OP-0001.

58.4 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), Sulfur cleanup (Method 3660), and Acid Cleanup (Method 3665). These cleanup procedures are included in SOP # PITT-OP-0001.

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59 SAFETY

59.1 Refer to section 5 of the Method 8000B SOP for general safety requirements.

59.2 Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

59.3 All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

59.4 All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

60 EQUIPMENT AND SUPPLIES

60.1 Refer to Section 6 of the 8000B section of this SOP. A GC equipped with a ^{63}Ni electron capture detector is required.

60.2 Refer to Table C-2 for analytical columns.

60.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

61 REAGENTS AND STANDARDS

61.1 Refer to the method 8000B section of this SOP for general requirements for reagents and supplies. All standards for this method must be replaced

61.2 Refer to Tables C-3 and C-6 for details of calibration standards.

61.3 Surrogate Standards

Tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) are the surrogate standards. Other surrogates may be used at client request. Refer to Table C-4 for details of surrogate standards.

62 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

63 QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

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64 PROCEDURE

64.1 Calibration and Standardization

64.2 Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

64.3 Initial Calibration

64.4 Refer to Table C-5 for the initial calibration analytical sequence.

64.5 The response for each Aroclor will be calculated by the procedures described in the general method for GC analysis, with the following modifications.

64.6 A five point calibration of the Aroclor 1016/1260 mix is generated plus at least a mid level single point standard for the other Aroclors. The average response factor is used to quantitate Aroclors 1260 and 1016. All other Aroclors are quantitated from the mid level single point standard.

64.7 The analyst may include a full 5 point calibration for any of the other Aroclors with the initial calibration.

64.8 The high and low standards for the initial 5 point calibration of Aroclors 1016/1260 define the acceptable quantitation range for the other Aroclors. If any Aroclor is determined above this concentration the extract must be diluted and reanalyzed.

64.9 If the analyst knows that a specific Aroclor is of interest for a particular project, that Aroclor may be used for the five point calibration rather than the Aroclor 1016/1260 mix.

64.10 The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

64.11 Two options are possible for quantitation of Aroclors. The same quantitation option must be used for standards and samples.

64.11.1.1 Multiple peak option: For each Aroclor, select 3-5 major peaks in the analyte pattern. Calculate the response factor of each major peak using the total area or total height of each peak. Calculate a %RSD using these response factors for each of the major peaks. Check each of these peaks to see if they pass the < 20% RSD criteria. To calculate the final concentration result in a sample, average the resultant concentrations from the 3-5 major peaks. When using this option, it is appropriate to remove peaks that appear to be co eluting with contaminate peaks from the quantitation. (I.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

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64.12 Initial Calibration Verification

64.12.1.1 The ICV will consist of second source standards of all Aroclors. Refer to the 8000B section of this SOP for acceptance criteria.

64.13 12 hour Calibration

64.13.1.1 The 12 hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration.

64.14 At a minimum, the 12 hour calibration includes analysis of the Aroclor 1016/1260 mix.

64.15 It is adequate to verify calibration with a mixture of Aroclors 1016/1260. If a specific Aroclor is expected, it should be included in the daily calibration check.

64.16 The retention time windows for any analytes included in the daily calibration are updated.

64.17 For this method samples must be bracketed with successful calibration verification runs.

64.18 Calibration verification

64.19 The Aroclor 1260/1016 calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.).

64.20 The daily CCV analysis, at a concentration other than the mid level (to meet NELAC requirements) will consist of Aroclors 1016/1260. All other CCVs will be a mid level Aroclor 1016/1260 standard.

64.21 A mid level calibration standard of the other five Aroclors (1221, 1232, 1242, 1248, 1254), to verify the initial calibration, is analyzed at least once every 72 hours.

Calibration Controls	Sequence	Control Limit
Initial Calibration Standards	6 pt. curve of AR1016/1260 prior to samples	≤20% RSD (alternatively, if the correlation coefficient is >0.99, linear regression may be used).

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Calibration Controls	Sequence	Control Limit
Second Source Verification	After initial calibration	± 20% Difference of expected value
Cont. Calib. Verif. (CCV)	After initial calibration, Every 20 samples	± 15% Difference*
Retention Time Windows	After initial calibration, update daily	3 X Std. Deviation
Note: If the CCV is > ±15% for any one compound, data will be acceptable if the result is J flagged. The result must be less than the RL.		

64.22 Procedure

64.23 Refer to the method 8000B section of this SOP for general procedural requirements.

64.24 Extraction

The extraction procedure is described in SOP No. PITT-OP-0001.

64.25 Cleanup

Cleanup procedures are described in SOP No. PITT-OP-0001.

64.26 An additional cleanup (Carboprep 90) may be performed prior to analysis if the sample extract has some color. Refer to Section 10.2.3.2 of this SOP for the Carboprep 90 Cleanup procedure.

64.27 Suggested gas chromatographic conditions are given in Table C-2.

64.28 Allow extracts to warm to ambient temperature before injection.

64.29 The suggested analytical sequence is given in Table C-5.

65 CALCULATIONS / DATA REDUCTION

65.1 Identification of Aroclors

Retention time windows are used for identification of Aroclors, but the “fingerprint” produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if

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in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

A clearly identifiable Aroclor pattern serves as confirmation of single column GC analysis. However, if the pattern is not clear, or if no historical data for the site is available, then second column confirmation must be performed.

65.2 Quantitation of Aroclors

Use 3-5 major peaks or total area for quantitation

All identifiable Aroclors in a sample will be reported. However, if more than one Aroclor is identified in a sample that have overlapping patterns, a discussion notifying the data user of these overlapping Aroclors will be included in the narrative.

65.3 If there are no interfering peaks within the envelope of the Aroclor, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

65.4 Second column confirmation of Aroclors will only be performed when requested by the client, or if the pattern is not clear or there is no historical data leading to a suspicion that Aroclors may be present. The appearance of the multi-peak "fingerprint" in the sample usually serves as a confirmation of Aroclor presence. Where second column confirmation is required for a project, refer to Section 11 of the main part of this SOP for details on confirmation procedures.

65.5 Surrogate recovery results are calculated and reported for tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) in all samples. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

66 METHOD PERFORMANCE

66.1 Performance limits for the four replicate initial demonstration of capability required under Section 12.2 of the main body of this SOP are recovery of 70-130% for Aroclors 1016/1260. The spiking level should be equivalent to a mid level calibration.

66.2 MDLs will be performed for Aroclors 1016/1260 annually. MDL verifications will be performed on all other Aroclors.

67 POLLUTION CONTROL

Refer to Section 13 of the 8000B section of this SOP.

68 WASTE MANAGEMENT

Refer to Section 14 of the 8000B section of this SOP.

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69 REFERENCES

69.1 Test Methods for Evaluating Solid Waste, SW-846, Update III, December 1996, Method 8082

69.2 SOP # PITT-QA-DoD-0001, Implementation of DoD QSM Version 3 January 2006, current version.

70 ATTACHMENTS

70.1 Table C-1: Standard Analyte List and Reporting Limits

70.2 Table C-2: Recommended GC Operating Conditions

70.3 Table C-3: Calibration Levels

70.4 Table C-4: LCS/Matrix Spike and Surrogate Spike Levels

70.5 Table C-5: Suggested Analytical Sequence

70.6 Table C-6: Preparation of Calibration Standards

70.7 Table C-7: Surrogate Recovery Limits

71 REVISION HISTORY

71.1 Revision 12, 09/07/2007

71.2 Changed the format of the SOP to correspond to the new Corporate SOP format.

72 METHOD MODIFICATIONS

72.1 Refer to section 18 of the 8000B section of this SOP.

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Table C-1				
Standard Analyte list and Reporting Limits				
Compound	**CAS #**	**Reporting Limit, µg/L, µg/wipe or µg/kg**		
		Water/Wipe Regular/Low Level	**Soil/Tissue Regular/Low Level**	**Waste**
Aroclor-1016	12674-11-2	0.4/0.01	16.67/0.833	500
Aroclor-1221	11104-28-2	0.4/0.01	16.67/0.833	500
Aroclor-1232	11141-16-5	0.4/0.01	16.67/0.833	500
Aroclor 1242	53469-21-9	0.4/0.01	16.67/0.833	500
Aroclor-1248	12672-29-6	0.4/0.01	16.67/0.833	500
Aroclor-1254	11097-69-1	0.4/0.01	16.67/0.833	500
Aroclor-1260	11096-82-5	0.4/0.01	16.67/0.833	500
Optional Compounds:				
Aroclor - 1262	37324-23-5	0.4/0.01	16.67/0.833	500
Aroclor - 1268	11100-14-4	0.4/0.01	16.67/0.833	500

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	**Final Vol.**	**Low Level Vol.**
Groundwater	1000 mL	40 mL	1 mL
Wipe	1 wipe	40 mL	NA
Low-Level Soil	15 g	20 mL	1 mL
High-Level Soil/Waste	1 g	40 mL	NA
Tissue	6 g	2 mL (1 mL with GPC clean-up)	NA

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Table C-2	
Parameter	**Recommended Conditions**
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	MR1, 30m x 0.53 mm id, 0.5µm
Column 2	MR2, 30m X 0.53 mm id, 0.5µm
Injection	1-2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table C-3							
Calibration Levels ng/mL							
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7[1]**
Aroclor 1016/1260	10	50	200	500	1000	2000	4000
Aroclor 1242[2]				500			
Aroclor 1221 +1254[2]				500			
Aroclor 1232[2]				500			
Aroclor 1248[2]				500			
Surrogates are included with all the calibration mixes at the following levels:							
Tetrachloro-m-xylene	0.5	2.5	10	25	50	100	200
Decachlorobiphenyl	0.5	2.5	10	25	50	100	200

[1] Level 7 is optional and should only be used if linearity can be maintained on the instrument to this level.

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[2] Aroclors may be quantitated within the range 100 to 2000 ng/mL (4000ng/mL if the level 6 1016/1260 standard is included). If the Aroclor is more concentrated, it must be reanalyzed at a dilution.

Table C-4

LCS/Matrix Spike and Surrogate Spike levels for Aroclor Analysis

µg/L, µg/wipe or µg/kg

	Aqueous/Wipe	Soil/Tissue	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table C-5

Suggested Analytical Sequence

Initial Calibration		
Injection No.		
1	Solvent Blank (optional)	Level 3
2	Aroclor 1221/1254	Level 3
3	Aroclor 1232	Level 3
4	Aroclor 1242	Level 3
5	Aroclor 1248	Level 3
6	Aroclor 1016/1260	Level 1
7	Aroclor 1016-1260	Level 2
8	Aroclor 1016-1260	Level 3
9	Aroclor 1016/1260	Level 4
10	Aroclor 1016/1260	Level 5
11	Aroclor 1016/1260	Level 6 (optional)

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Table C-5		
Suggested Analytical Sequence		
12-16	ICVs (second source standard of all Aroclors)	
17	Solvent Blank	
18-37	Sample 1-20 (or 12 hours)	
38	Aroclor 1016/1260	Level 3, etc.
12-Hour Calibration		
At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Aroclor 1016/1260 mix. Mid level standards of any other Aroclors expected to be present in the samples are also injected.		

Table C-6					
Preparation of Calibration Standards					
Calibration Level	**1016/1260 Intermediate (uL)**	**1221 + 1254 Stock (uL)**	**1232 Stock (uL)**	**1242 Stock (uL)**	**1248 Stock (uL)**
Level 1	4				
Level 2	20				
Level 3	80				
Level 4	1000	250	250	250	250
Level 5	400				
Level 6	800				
Level 7	1600				

The surrogate stock is purchased (Decachlorobiphenyl and Tetrachloro-m-xylene) at 200 ug/mL.

The Aroclor 1016 and 1260 stock standards are purchased as certified standards in isooctane at 1000 ug/mL. The other five Aroclor stock standards are purchased at 200 ug/mL.

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For Aroclors 1016 and 1260, an intermediate standard is prepared by diluting 1.0 mL of each of the stock standards and 0.25 mL of the surrogate stock standard to 10.0 mL in hexane. The intermediate Aroclor 1016/1260 standard concentrations are 100 ug/mL for each Aroclor and 5 ug/mL for each surrogate.

The Aroclor 1016/1260 calibration standards are prepared by diluting the volumes noted in Table C-6 to a 40.0 mL final volume in hexane except for the Level 3 standard, which is taken to a 200 mL final volume in hexane.

The mid level (Level 3) calibration standards for each of the other five Aroclors (1221, 1232, 1242, 1248, 1254) are prepared by diluting 0.25 mL of the appropriate stock standard to a final volume of 100 mL in hexane. Aroclors 1221 and 1254 are combined into one standard and Aroclors 1232, 1242, and 1248 are prepared individually.

Table C-7

Surrogate Recovery Limits

Surrogate	Water Recovery Limits	Soil Recovery Limits
Decachlorobiphenyl	24-128	23-141
Tetrachloro-m-xylene	45-120	31-127

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Appendix D

Analysis of Organophosphorus Pesticides Based on Method 8141A

73 SCOPE AND APPLICATION

73.1 This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of organophosphorous pesticides by GC/FPD. This Appendix is to be applied when SW-846 Method 8141A is requested, and is applicable to extracts derived from any matrices, which are prepared according to the appropriate TestAmerica sample extraction SOPs. (PITT-OP-0001)

73.2 Table D-1 lists compounds, which are routinely determined by this method and their associated Reporting Limits (RL) for each matrix. RLs given are based on the low-level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

74 SUMMARY OF METHOD

74.1 This method presents conditions for the analysis of prepared extracts for organophosphorous pesticides. The pesticides are injected onto the GC column and separated and detected by Flame Photometric detection. Quantitation is by the external standard method.

75 DEFINITIONS

75.1 Refer to the LQM for definitions of terms used in this document.

76 INTERFERENCES

76.1 Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

76.2 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the flame photometric detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding sample/reagent contact with plastic materials.

76.3 Sulfur will interfere and can be removed using procedures described in SOP PITT-OP-0001.

76.4 Interferences extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups have not been determined for this method.

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77 SAFETY

77.1 Refer to section 5 of the Method 8000B SOP for general safety requirements.

78 EQUIPMENT AND SUPPLIES

78.1 Refer to Section 6 of the 8000B section of this SOP. A GC equipped with a flame photometric detector is required.

78.2 Refer to Table D-2 for analytical columns.

78.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

79 REAGENTS AND STANDARDS

79.1 Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

79.2 Refer to Tables D-3 and D-8 for details of calibration standards.

79.3 Surrogate Standards

Triphenyl phosphate and Tributyl phosphate are the surrogate standards. Refer to Table D-4 for details of surrogate standards.

80 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

80.1 Refer to Section 8 of the 8000B section of this SOP.

81 QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

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82 PROCEDURE

82.1 Calibration and Standardization

82.2 Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

82.3 Refer to Table D-2 for details of GC operating conditions.

82.4 Initial Calibration

82.5 Refer to Section 10 of the 8000B section of this SOP for details of calibration procedures.

82.6 A five point calibration of each of the individual analyte mixes is generated. Up to 2 separate mixes (A, and B) are prepared to ensure that there is complete resolution of all analytes in the mixes.

82.7 The surrogate calibration curve is calculated from the Mix A. Surrogates in the other calibration standards are used only as retention time markers.

82.8 Refer to Table D-7 for the initial calibration analytical sequence.

82.9 The response for each single-peak analyte will be calculated by the procedures described in the general method for GC analysis.

82.10 Initial Calibration Verification

82.10.1.1 The ICV will consist of second source standards of all analytes of interest. Refer to the 8000B section of this SOP for acceptance criteria.

82.11 12 hour Calibration Verification

82.11.1.1 The 12 hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration. A mid level calibration standard is analyzed every 10 samples. Refer to the 8000B section of this SOP for acceptance criteria.

82.11.1.2 The retention time windows for any analytes included in the 12 hour calibration are updated.

82.12 Continuing Calibration

82.13 The mid-level calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after **every 10 samples**, including matrix spikes, LCS, and method blanks. If 12 hours elapse analyze the 12 hour standard sequence instead. If

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instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

82.14 The daily CCV analysis at a concentration other than the mid level (to meet NELAC requirements) will consist of all of the analytes of interest. All other CCVs will be mid level calibration standards.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5 pt. Curve (minimum)	≤ 20% RSD (alternatively, if the correlation coefficient is >0.99, linear regression may be used).
Second Source	prior to samples	± 20 % of expected value
Retention Time Windows	after calibration, update daily	3 X Standard Deviation
Cont. Cal. Verif. (CCV)	every **10** samples	± 15% D
Note: If the CCV is > ±15% for any one compound, data will be acceptable if the result is J flagged. The result must be less than the RL.		

82.15 Procedure

82.16 Refer to the method 8000B section of this SOP for general procedural requirements.

82.17 Extraction - The extraction procedure is described in SOP No. PITT-OP-0001.

82.18 Cleanup - Cleanup procedures are described in SOP No. PITT-OP-0001.

82.19 Suggested gas chromatographic conditions are given in Table D-2.

82.20 Allow extracts to warm to ambient temperature before injection.

82.21 The suggested analytical sequence is given in Table D-7.

83 CALCULATIONS / DATA REDUCTION

83.1 Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

83.2 Surrogate recovery results are calculated and reported for Triphenyl phosphate and Tributylphosphate. Corrective action is only necessary if Triphenyl phosphate and Tributyl phosphate are both outside of acceptance limits.

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84 METHOD PERFORMANCE

84.1 Current laboratory performance limits are listed in Tables D-5 and D-6. The spiking level should be equivalent to a mid-level calibration.

85 POLLUTION CONTROL

Refer to Section 13 of the 8000B section of this SOP.

86 WASTE MANAGEMENT

86.1 Refer to Section 14 of the 8000B section of this SOP.

87 REFERENCES

87.1 Test Methods for Evaluating Solid Waste, SW-846, Update III, December 1996, Method 8141A

87.2 SOP # PITT-QA-DoD-0001, Implementation of DoD QSM Version 3 January 2006, current version.

88 ATTACHMENTS

88.1 Table D-1: Standard Analyte List and Reporting Limits

88.2 Table D-2: Recommended GC Operating Conditions

88.3 Table D-3: Calibration Levels

88.4 Table D-4: LCS/Matrix Spike and Surrogate Spike Levels

88.5 Table D-4A: LCS and Matrix Spike Control Limits

88.6 Table D-5: Aqueous Acceptance Criteria for Initial Demonstration of Capability

88.7 Table D-6: Solid Acceptance Criteria for Initial Demonstration of Capability

88.8 Table D-7: Suggested Analytical Sequence

88.9 Table D-8: Surrogate Recovery Limits

88.10 Table D-9: Preparation of Calibration Standards

89 REVISION HISTORY

89.1 Revision 12, 09/07/2007

89.2 Changed the format of the SOP to correspond to the new Corporate SOP format.

90 METHOD MODIFICATIONS

90.1 Refer to section 18 of the 8000B section of this SOP.

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Table D-1

Standard Analyte list and Reporting Limits

Compound	CAS #	Reporting Limit, µg/L, µg/wipe or µg/kg	
		Water/Wipe	Soil
Dimethoate	60-51-5	1	33
Disulfoton	298-04-4	1	33
Famphur	52-85-7	1	33
Methyl parathion	298-00-0	1	33
O,O,O-Triethyl phosphorothioate	126-68-1	1	33
Parathion (Ethyl parathion)	56-38-2	1	33
Phorate	298-02-2	1	33
Tetraethyldithiopyrophosphate (Sulfotepp)	3689-24-5	1	33
Thionazin	297-97-2	1	33
Azinphos-methyl (Guthion)	86-50-0	1	33
Bolstar (Sulprofos)	35400-43-2	1	33
Chlorpyrifos (Dursban)	2921-88-2	1	33
Coumaphos	56-72-4	1	33
Demeton-O	298-03-3	1	33
Dementon-S	126-75-0	1	33
Demeton (total)	8065-48-3	1	33
Diazinon	333-41-5	1	33
Dichlorvos	62-73-7	1	33
EPN	2104-64-5	1	33
Ethoprop (Mocap)	13194-48-4	1	33

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Table D-1			
Standard Analyte list and Reporting Limits			
Compound	**CAS #**	**Reporting Limit, µg/L, µg/wipe or µg/kg**	
		Water/Wipe	**Soil**
Fensulfothion	115-90-2	1	33
Fenthion	55-38-9	1	33
Malathion	121-75-5	1	33
Mevinphos (Phosdrin)	7786-34-7	1	33
Ronnel	299-84-3	1	33
Stirophos	961-11-5	1	33
Tokuthion	53-10-0	1	33
Trichloronate	327-98-0	1	33

Table D-2	
Parameter	**Recommended Conditions**
Injection port temp	220°C
Detector temp	250°C
Temperature program	110C for .5 min, 3.0°C/min to 250°C, 2.84 min hold
Column 1	RTX OPP 30m x 0.32mm id, 0.5µm
Column 2	RTX OPP2 30m x 0.32 mm id, 0.32µm
Injection	2µL
Carrier gas	Helium
Make up gas	Helium

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Table D-3					
Calibration Levels ng/mL					
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**
Dimethoate	.2	.5	1.0	2.0	4.0
Disulfoton	.2	.5	1.0	2.0	4.0
Famphur	.2	.5	1.0	2.0	4.0
Methyl parathion	.2	.5	1.0	2.0	4.0
O,O,O-Triethyl phosphorothioate	.2	.5	1.0	2.0	4.0
Parathion (Ethyl parathion)	.2	.5	1.0	2.0	4.0
Phorate	.2	.5	1.0	2.0	4.0
Tetraethyldithiopyrophosphate (Sulfotepp)	.2	.5	1.0	2.0	4.0
Thionazin	.2	.5	1.0	2.0	4.0
Tributyl phosphate (surrogate)	.2	.5	1.0	2.0	4.0
Triphenyl phosphate (surrogate)	.2	.5	1.0	2.0	4.0
Azinphos-methyl	.2	.5	1.0	2.0	4.0
Bolstar	.2	.5	1.0	2.0	4.0
Chlorpyrifos	.2	.5	1.0	2.0	4.0
Coumaphos	.2	.5	1.0	2.0	4.0
Demeton (total) *	.2	.5	1.0	2.0	4.0
Diazinon	.2	.5	1.0	2.0	4.0
Dichlorvos	.2	.5	1.0	2.0	4.0
EPN	.2	.5	1.0	2.0	4.0
Ethoprop	.2	.5	1.0	2.0	4.0
Fensulfothion	.2	.5	1.0	2.0	4.0
Fenthion	.2	.5	1.0	2.0	4.0

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Table D-3					
Calibration Levels ng/mL					
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**
Malathion	.2	.5	1.0	2.0	4.0
Mevinphos	.2	.5	1.0	2.0	4.0
Ronnel	.2	.5	1.0	2.0	4.0
Stirophos	.2	.5	1.0	2.0	4.0
Tokuthion	.2	.5	1.0	2.0	4.0
Trichloronate	.2	.5	1.0	2.0	4.0

1 Standards may be split into multiple mixes if resolution of all compounds on both columns is not obtained.

Note: Component mixes of a CCAL should be run sequentially. The CCAL evaluation is performed on the sum of the mixes, rather than by mix. I.e. the CCAL = sum of the component mixes.

***** Demeton is a mixture of two compounds; Demeton-O and Demeton-S; therefore will elute as two peaks.

Table D-4		
LCS/Matrix Spike and Surrogate Spike levels µg/L, µg/wipe or µg/kg		
	Aqueous/Wipe	**Soil**
Dimethoate[1]	10	333
Disulfoton[1]	10	333
Famphur[1]	10	333
Methyl parathion[1]	10	333
O,O,O-Triethyl phosphorothioate[1]	10	333
Parathion (Ethyl parathion)[1]	10	333
Phorate[1]	10	333

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Table D-4		
LCS/Matrix Spike and Surrogate Spike levels µg/L, µg/wipe or µg/kg		
	Aqueous/Wipe	**Soil**
Tetraethyldithiopyrophosphate (Sulfotepp) [1]	10	333
Thionazin[1]	10	333
Azinphos-methyl	10	333
Bolstar	10	333
Chlorpyrifos	10	333
Coumaphos	10	333
Demeton (total)*	10	333
Diazinon	10	333
Dichlorvos	10	333
EPN	10	333
Ethoprop	10	333
Fensulfothion	10	333
Fenthion	10	333
Malathion	10	333
Mevinphos	10	333
Ronnel	10	333
Stirophos	10	333
Tokuthion	10	333
Trichloronate	10	333
Tributyl phosphate (surrogate)	10	333
Triphenyl phosphate (surrogate)	10	333

[1] Typical spike list contains these compounds.

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Table D4A

LCS and Spike Control Limits

	Soil						Water					
	LCS			Spike			LCS			Spike		
Compound	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD
Azinphos-methyl	10	150	35	10	150	35	10	150	35	10	150	35
Bolstar	10	150	35	10	150	35	10	150	35	10	150	35
Chlorpyrifos	50	130	25	50	130	25	50	130	25	50	130	25
Coumaphos	10	150	35	10	150	35	10	150	35	10	150	35
Demeton (total)	10	150	35	10	150	35	10	150	35	10	150	35
Demeton-O	10	150	35	10	150	35	10	150	35	10	150	35
Demeton-S	10	150	35	10	150	35	10	150	35	10	150	35
Diazinon	10	150	35	10	150	35	10	150	35	10	150	35
Dichlorvos	10	150	35	10	150	35	10	150	35	10	150	35
Dimethoate	51	140	35	37	140	35	26	136	22	26	136	22
Disulfoton	31	136	33	10	139	33	36	124	46	20	138	46
EPN	10	150	35	10	150	35	10	150	35	10	150	35
Ethoprop	10	150	35	10	150	35	10	150	35	10	150	35
Ethyl parathion	52	133	38	40	133	38	56	125	21	48	127	21
Famphur	54	137	30	50	137	30	52	131	24	47	136	24
Fensulfothion	10	150	35	10	150	35	10	150	35	10	150	35
Fenthion	10	150	35	10	150	35	10	150	35	10	150	35
Malathion	10	150	35	10	150	35	10	150	35	10	150	35
Methyl parathion	43	146	41	35	146	41	41	148	20	41	148	20
Mevinphos	10	150	35	10	150	35	10	150	35	10	150	35
O,O,O-Triethyl phosphorothioate	20	127	36	11	127	36	31	131	61	15	131	61

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Table D4A

LCS and Spike Control Limits

	Soil						Water					
	LCS			Spike			LCS			Spike		
Compound	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD
Parathion	52	133	38	40	133	38	56	125	21	48	127	21
Phorate	41	143	43	26	134	43	41	135	24	39	135	24
Ronnel	10	150	35	10	150	35	10	150	35	10	150	35
Stirophos	10	150	35	10	150	35	10	150	35	10	150	35
Sulfotepp	48	126	28	42	121	28	56	114	23	46	116	23
Tetraethyldithiopyrophosphate	48	126	28	42	121	28	56	114	23	46	116	23
Thionazin	48	126	27	48	165	27	44	128	21	44	128	21
Tokuthion	10	150	35	10	150	35	10	150	35	10	150	35
Tributyl phosphate	55	125	0	55	125	0	49	122	0	49	122	0
Trichloronate	10	150	35	10	150	35	10	150	35	10	150	35
Triphenyl phosphate	47	130	0	47	130	0	45	145	0	45	145	0

Table D-5

Aqueous Acceptance Criteria for Initial Demonstration of Capability

		Control Limits	
Compound	ug/L	LCL	UCL
Dimethoate	10	46	153
Disulfoton	10	17	185
Famphur	10	14	165
Methyl parathion	10	36	159

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Table D-5			
Aqueous Acceptance Criteria for Initial Demonstration of Capability			
		Control Limits	
Compound	**ug/L**	**LCL**	**UCL**
O,O,O-Triethyl phosphorothioate	10	65	141
Parathion	10	13	150
Phorate	10	35	152
Tetraethyldithiopyrophosphate	10	75	140
Thionazin	10	69	149
Tributyl phosphate (surrogate)	10	30	150
Triphenyl phosphate (surrogate)	10	25	152

Table D-6			
Solid Acceptance Criteria for Initial Demonstration of Capability			
		Control Limits	
Compound	**ug/kg**	**LCL**	**UCL**
Dimethoate	333	65	135
Disulfoton	333	66	133
Famphur	333	42	162
Methyl parathion	333	64	144
O,O,O-Triethyl phosphorothioate	333	58	131
Parathion	333	58	141
Phorate	333	71	135
Tetraethyldithiopyrophosphate	333	69	144

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Table D-6			
Solid Acceptance Criteria for Initial Demonstration of Capability			
		Control Limits	
Compound	**ug/kg**	**LCL**	**UCL**
Thionazin	333	68	140
Tributyl phosphate (surrogate)	333	30	150
Triphenyl phosphate (surrogate)	333	20	151

Table D-7	
Suggested Analytical Sequence	
Initial Calibration	
Solvent Blank (optional)	
Calibration Mix A	All Levels
Calibration Mix B	All Levels
ICV (second source standards)	
Solvent Blank	
Up to 20 samples (unless 12 hours comes first)	
Solvent Blank (optional)	
Individual A Mix	Mid-Level
Individual B Mix	Mid-Level
Up to 20 samples (unless 12 hours comes first)	
Individual A Mix	Mid-Level
Individual B Mix	Mid-Level
After 12 hours	
Individual A Mix	Mid-Level

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Table D-7	
Suggested Analytical Sequence	
Individual B Mix	Mid-Level
Up to 20 samples (unless 12 hours comes first)	
Individual A Mix	Mid-Level
Individual B Mix	Mid-Level
12-Hour Calibration	
At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Individual mixes A, B, and C.	

Table D-8		
Surrogate Recovery Limits		
Surrogate	**Water Recovery Limits**	**Soil Recovery Limits**
Triphenyl Phosphate	45-145	47-130
Tributyl Phosphate	49-122	55-125

Table D-9		
Preparation of Calibration Standards		
Calibration Level	**Mix A Stock (uL)**	**Mix B Stock (uL)**
Level 1	50	50
Level 2	200	200
Level 3	400	400

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Table D-9		
Preparation of Calibration Standards		
Calibration Level	**Mix A Stock (uL)**	**Mix B Stock (uL)**
Level 4	800	800
Level 5	1000	1000

The Mix A and Mix B stock standards are purchased as certified standards in two separate solutions in 95%hexane/5% acetone at 100 ug/mL for each analyte. The Mix A stock includes Demeton, Dichlorvos, EPN, Fenthion, Azinphos-methyl, Methyl parathion, Ethoprop, Stirofos, Tokuthion, Tributyl phosphate (surrogate) and Triphenyl phosphate (surrogate). The Mix B stock includes Coumaphos, Diazinon, Dimethoate, Disulfoton, Chlorpyrifos, Famfur, Fensulfothion, Malathion, O,O,O-Triethylphosphorothioate, Parathion, Phorate, Melvinphos, Ronnel, Sulfotep, Bolstar, Thionazin, and Trichloronate. The working Mix A and Mix B calibration standards are prepared by diluting the volume of each stock noted in Table D-8 to a 25 mL final volume in hexane for the Level 1 and Level 5 standards, and to a 40 mL final volume for the Level 2, Level 3, and Level 4 standards.

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Appendix E

Analysis of PAH's Based on Method 610 and 8310

91 SCOPE AND APPLICATION

This SOP Appendix describes procedures to be used when SW-846 Method 8000B or method 610 is applied to the analysis of Polynuclear Aromatic Hydrocarbons (PAHs) by High Performance Liquid Chromatography (HPLC). This Appendix is to be applied when SW-846 Method 8310 or 610 is requested. Method 8310 is applicable to extracts derived from any matrix, which are prepared according to the appropriate TestAmerica sample extraction SOPs. (PITT-OP-0001) Method 610 is only applicable to water samples.

Table E-1 lists compounds, which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

92 SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts for Polynuclear Aromatic Hydrocarbons (PAHs). The extracts are injected onto the HPLC column and separated and detected by ultraviolet (UV) and fluorescence detection. Quantitation is by the external standard method.

93 DEFINITIONS

Refer to the LQM for definitions of terms used in this document.

94 INTERFERENCES

94.1 Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

94.2 Interferences in the HPLC analysis arise from many compounds amenable to HPLC that give a measurable response on the UV and/or fluorescence detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

94.3 Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples.

95 SAFETY

95.1 Refer to section 5 of the Method 8000B SOP for general safety requirements.

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96 EQUIPMENT AND SUPPLIES

96.1 Refer to Section 6 of the 8000B section of this SOP. A HPLC equipped with both UV and fluorescence detectors are required.

96.2 Refer to Table E-2 for analytical columns.

96.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

97 REAGENTS AND STANDARDS

97.1 Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

97.2 All standards are purchased as stock solutions, but may be optionally prepared from single ampules or neat. Secondary dilution standards (prepared from stock) must be prepared every six months or sooner, if standards have degraded or concentrated. Refer to Tables E-3, E-8, and E-9 for details of calibration standards.

97.3 Acetonitrile; pesticide grade or equivalent.

Benzo(e)pyrene and p-terphenyl are the surrogate standards. Refer to Tables E-4 for details of surrogate standards.

Super-Q and/or HPLC grade water.

98 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

99 QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

For method 610, a blank, LCS and matrix spike must be performed every 10 samples.

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100 PROCEDURE

100.1 Calibration and Standardization

100.2 Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

100.3 Refer to Table E-2 for details of HPLC operating conditions. The conditions listed should result in resolution of all analytes listed in Table E-1 on both detectors.

100.4 Recommended calibration levels are given in Table E-3.

100.5 The compound acenaphthylene only responds on the UV detector, therefore can only be reported from the UV detector. For DoD work acenapthylene can be confirmed on the UV detector using a wavelength at 227 nm.

100.6 Initial Calibration

100.7 Refer to Section 10 of the 8000B section of this SOP for details of calibration procedures.

100.8 Refer to Table E-7 for the initial calibration analytical sequence.

100.9 The response for each single-peak analyte will be calculated by the procedures described in the 8000B section of this SOP. For method 610 the % RSD must be < 10%.

100.10 Initial Calibration Verification

100.11 The ICV will consist of a second source standard of all analytes of interest. Refer to the 8000B section of this SOP for acceptance criteria.

100.12 12 hour Calibration Verification

100.13 The 12 hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration. A mid level calibration standard is used for the 12 hour calibration. Refer to the 8000B section of this SOP for acceptance criteria.

100.14 The retention time windows for any analytes included in the 12 hour calibration are updated.

100.15 Continuing Calibration

100.16 The mid-level calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. If 12 hours elapse analyze the 12 hour standard sequence instead. If

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instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

100.17 The daily CCV analysis at a concentration other than the mid level (to meet NELAC requirements) will contain all analytes of interest. All other CCVs will be mid level calibration standards.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5 pt. Curve (minimum)	≤ 20% RSD – 8310 (alternatively, if the correlation coefficient is >0.99, linear regression may be used).
Second Source	prior to samples	± 20 % of expected value
Retention Time Windows	after calibration, update daily	3 X Standard Deviation
Cont. Cal. Verif. (CCV)	every 20 samples	± 15% D
Note: If the CCV is > ±15% for any one compound, data will be acceptable if the result is J flagged. The result must be less than the RL.		

100.18 Procedure

100.19 Refer to the method 8000B section of this SOP for general procedural requirements.

100.20 Extraction

The extraction procedure is described in SOP No. PITT-OP-0001.

100.21 Cleanup

The cleanup procedures are described in SOP No. PITT-OP-0001.

100.22 Suggested HPLC conditions are given in Table E-2.

100.23 Allow extracts to warm to ambient temperature before injection.

100.24 The suggested analytical sequence is given in Table E-7.

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101 CALCULATIONS / DATA REDUCTION

101.1 Refer to Section 11 of the 8000B section of this SOP for compound identification and quantitation.

101.2 The compound acenaphthylene only responds on the UV detector, therefore can only be reported from the UV detector. For DoD work acenapthylene can be confirmed on the UV detector using a wavelength at 227 nm.

101.3 Surrogate recovery results are calculated and reported for Benzo(e)pyrene and p-terphenyl. Corrective action is only necessary if Benzo(e)pyrene and p-terphenyl are both outside of acceptance limits.

102 METHOD PERFORMANCE

102.1 Performance limits for the four replicate initial demonstration of capability required under Section 12.2 of the main body of this SOP are presented in Tables E-5 and E-6. The spiking level should be equivalent to a mid level calibration.

103 POLLUTION CONTROL

Refer to Section 13 of the 8000B section of this SOP.

104 WASTE MANAGEMENT

104.1 Refer to Section 14 of the 8000B section of this SOP.

105 REFERENCES

105.1 Test Methods for Evaluating Solid Waste, SW-846, Update III, December 1996, Method 8310

105.2 SOP # PITT-QA-DoD-0001, Implementation of DoD QSM Version 3 January 2006, current version.

106 ATTACHMENTS

106.1 Table E-1: Standard Analyte List and Reporting Limits

106.2 Table E-2: Recommended GC Operating Conditions

106.3 Table E-3: Calibration Levels

106.4 Table E-4: LCS/Matrix Spike and Surrogate Spike Levels

106.5 Table E-5: Aqueous LCS and Matrix Spike Control Limits

106.6 Table E-6: Solid LCS and Matrix Spike Control Limits

106.7 Table E-7: Suggested Analytical Sequence

106.8 Table E-8: Stock Standard Concentrations

106.9 Table E-9: Preparation of Calibration Standards

106.10 Table E-10: Surrogate Recovery Limits

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107 REVISION HISTORY

107.1 Revision 12, 09/07/2007

107.2 Changed the format of the SOP to correspond to the new Corporate SOP format.

108 METHOD MODIFICATIONS

108.1 Refer to Section 18 of the 8000B section of this SOP.

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Table E-1

Standard Analyte list and Reporting Limits and Detectors

Compound	CAS #	Reporting Limit, µg/L or µg/kg		
		Water/Low-Level Water	Soil or Tissue	Detectors
Carbazole	86-74-8	1.0 / 0.2	33	UV/Fluorescence
Naphthalene	91-20-3	1.0 / 0.2	33	UV/Fluorescence
Acenaphthene	83-32-9	1.0 / 0.2	33	UV/Fluorescence
Acenaphthylene *	208-96-8	1.0 / 0.2	33	UV
Anthracene	120-12-7	.2 / 0.04	6.7	UV/Fluorescence
Benzo(a)anthracene	56-55-3	.2 / 0.04	6.7	UV/Fluorescence
Benzo(b)fluoranthene	205-99-2	.2 / 0.04	6.7	UV/Fluorescence
Benzo(k)fluoranthene	207-08-9	.2 / 0.04	6.7	UV/Fluorescence
Benzo(g,h,i)perylene	191-24-2	.2 / 0.04	6.7	UV/Fluorescence
Benzo(a)pyrene	50-32-8	.2 / 0.04	6.7	UV/Fluorescence
Chrysene	218-01-9	.2 / 0.04	6.7	UV/Fluorescence
Fluoranthene	206-44-0	.2 / 0.04	6.7	UV/Fluorescence
Fluorene	86-73-7	.2 / 0.04	6.7	UV/Fluorescence
Indeno(1,2,3-cd)pyrene	193-39-5	.2 / 0.04	6.7	UV/Fluorescence
Pyrene	129-00-0	.2 / 0.04	6.7	UV/Fluorescence
Phenanthrene	85-01-8	.2 / 0.04	6.7	UV/Fluorescence
Dibenzo(a,h)anthracene	53-70-3	.2 / 0.04	6.7	UV/Fluorescence
1-methylnaphthalene	90-12-0	1.0 / 0.2	33	UV/Fluorescence
2-methylnaphthalene	91-57-6	1.0 / 0.2	33	UV/Fluorescence

* Acenaphthylene only responds on the less selective UV detector and therefore is not confirmed by second detector using this procedure. This compound may be prone to false positives in complex matrices (i.e., samples with high concentrations of petroleum or other interferences; tissues; etc.). Other methods of determination (i.e.,

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GC/MS or GC/MS SIM) should be considered for this compound if the matrix is known to contain high concentrations of co-extracting interferents. For DoD work acenapthylene can be confirmed on the UV detector using a wavelength at 227 nm.

The following concentration factors are assumed in calculating the Reporting Limits:

	Final Vol.	**Extraction Vol.**
Low-Level Goundwater	1000 mL	1.0 mL
Goundwater	1000 mL	5.0 mL
Low-Level Soil	15 g	0.5 mL
High-Level Soil/Waste	1 g	1.0 mL
Tissue	30 g	0.5 mL (with GPC clean-up)

Table E-2	
Parameter	**Recommended Conditions**
Mobile phase program	Time 0min:solvent A 50% solvent B 50% flow 1.5 ml/min Time 7min:solvent A 65% solvent B 35% flow 1.5 ml/min Time 10min:solvent A 75% solvent B 25% flow 1.5 ml/min Time 15min:solvent A 85% solvent B 15% flow 1.5 ml/min Time 20min:solvent A 95% solvent B 5% flow 1.5 ml/min
Column 1	Hypersil PAH 150mm x 4.6mm ID
Injection	20µL
UV Signal: Fluorescence Detector Excitation/Emission:	254 nm Excitation: 220-380 nm Emission: 300 –500 nm
Solvent A	CH3CN
Solvent B	H2O

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Table E-3							
Calibration Levels ng/mL							
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**
Individual Mix							
Carbazole	0.2	0.5	1.0	5.0	10	20	40
Naphthalene	0.2	0.5	1.0	5.0	10	20	40
Acenaphthene	0.2	0.5	1.0	5.0	10	20	40
Acenaphthylene	0.2	0.5	1.0	5.0	10	20	40
Anthracene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Benzo(a)anthracene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Benzo(b)fluoranthene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Benzo(k)fluoranthene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Benzo(g,h,i)perylene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Benzo(a)pyrene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Chrysene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Fluoranthene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Fluorene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Indeno(1,2,3-cd)pyrene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Pyrene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Phenanthrene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
Dibenzo(a,h)anthracene	0.04	0.1	0.2	1.0	2.0	4.0	8.0
1-methylnaphthalene	0.2	0.5	1.0	5.0	10	20	40
2-methylnaphthalene	0.2	0.5	1.0	5.0	10	20	40
Surrogates							
p-terphenyl	0.2	0.5	1.0	5.0	10	20	40
Benzo(e)pyrene	0.2	0.5	1.0	5.0	10	20	40

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Table E-4		
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg		
	Aqueous	**Soil/Tissue**
Carbazole	25.0	416.67
Naphthalene	25.0	416.67
Acenaphthene	25.0	416.67
Acenaphthylene	25.0	416.67
Anthracene	5.0	83.33
Benzo(a)anthracene	5.0	83.33
Benzo(b)fluoranthene	5.0	83.33
Benzo(k)fluoranthene	5.0	83.33
Benzo(g,h,i)perylene	5.0	83.33
Benzo(a)pyrene	5.0	83.33
Chrysene	5.0	83.33
Fluoranthene	5.0	83.33
Fluorene	5.0	83.33
Indeno(1,2,3-cd)pyrene	5.0	83.33
Pyrene	5.0	83.33
Phenanthrene	5.0	83.33
Dibenzo(a,h)anthracene	5.0	83.33
1-methylnaphthalene	25.0	416.67
2-methylnaphthalene	25.0	416.67
p-terphenyl (surrogate)	20.0	666.67
Benzo(e)pyrene (surrogate)	10.0	333.3

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Table E-5

Aqueous LCS and Matrix Spike Acceptance Criteria

	LCS			Matrix Spike		
Compound	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
Acenaphthene	37	135	27	20	144	27
Acenaphthylene	42	132	20	22	147	25
Anthracene	60	118	20	29	147	25
Benzo(a)anthracene	62	133	20	57	135	25
Benzo(b)fluoranthene	68	133	20	64	133	25
Benzo(k)fluoranthene	67	122	20	64	122	25
Benzo(ghi)perylene	65	128	20	65	128	25
Benzo(a)pyrene	62	120	20	60	120	25
Carbazole	50	125	25	50	125	25
Chrysene	67	129	20	67	130	25
Dibenzo(a,h)anthracene	65	126	20	65	126	25
Fluoranthene	57	126	20	46	146	25
Fluorene	47	128	20	24	157	25
Indeno(1,2,3-cd)pyrene	71	124	20	52	128	25
2-Methylnaphthalene	50	125	25	50	125	25
1-Methylnaphthalene	50	125	25	50	125	25
Naphthalene	41	128	20	10	153	25
Phenanthrene	46	141	20	33	162	25
Pyrene	56	129	20	44	139	25
Benzo(e)pyrene	78	125		78	125	
Terphenyl-d14	72	125		72	125	

Note: The limits are based on laboratory-generated data.

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Table E-6

Solid LCS and Matrix Spike Acceptance Criteria

	LCS			Matrix Spike		
Compound	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
Acenaphthene	19	122	27	29	144	27
Acenaphthylene	20	140	20	20	147	25
Anthracene	19	120	20	19	147	25
Benzo(a)anthracene	50	137	20	50	137	25
Benzo(b)fluoranthene	63	134	50	28	162	50
Benzo(k)fluoranthene	63	127	26	43	140	26
Benzo(ghi)perylene	56	134	20	56	134	25
Benzo(a)pyrene	63	121	25	32	126	25
Carbazole	50	125	25	50	125	25
Chrysene	55	135	33	38	128	33
Dibenzo(a,h)anthracene	65	124	28	38	173	28
Fluoranthene	28	141	25	28	114	25
Fluorene	22	134	20	22	157	25
Indeno(1,2,3-cd)pyrene	58	133	33	35	141	33
2-Methylnaphthalene	50	125	25	50	125	25
1-Methylnaphthalene	50	125	25	50	125	25
Naphthalene	15	118	20	10	153	25
Phenanthrene	20	138	48	20	140	48
Pyrene	49	124	20	44	139	25
Benzo(e)pyrene	49	129		49	129	
Terphenyl-d14	54	126		54	126	

Note: The limits are based on laboratory-generated data.

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Table E-7	
Suggested Analytical Sequence	
Initial Calibration	Levels 1 through 7
ICV (second source standard)	
Up to 20 samples (unless 12 hours comes first)	
CCV (alternating levels)	Medium Level
Up to 20 samples (unless 12 hours comes first)	
CCV (alternating levels)	Medium-High Level
Up to 20 samples (unless 12 hours comes first)	
CCV (alternating levels)	Medium Level
Up to 20 samples (unless 12 hours comes first)	
CCV (alternating levels)	Medium-Low Level
Up to 20 samples (unless 12 hours comes first)	
CCV (alternating levels)	Medium Level
Retention time windows are updated initially from the mid-level standard of the initial calibration and should be updated every 24-hours thereafter from the medium-level CCV, but can be updated more frequently if the instrument shows signs of drift.	

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Table E-8	
Stock Standard Concentrations (ug/mL)	
Carbazole	500
Naphthalene	500
Acenaphthene	500
Acenaphthylene	500
Anthracene	100
Benzo(a)anthracene	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(g,h,i)perylene	100
Benzo(a)pyrene	100
Chrysene	100
Fluoranthene	100
Fluorene	100
Indeno(1,2,3-cd)pyrene	100
Pyrene	100
Phenanthrene	100
Dibenzo(a,h)anthracene	100
1-methylnaphthalene	500
2-methylnaphthalene	500
p-terphenyl (surrogate)	500
Benzo(e)pyrene (surrogate)	500

The stock standard is a certified solution in acetonitrile.

Note: Alternate approaches to standard preparation may be taken, including alternate stock standard concentrations, and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations are maintained.

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Table E-9		
Preparation of Calibration Standards		
Calibration Level	**Volume of Stock Std. (uL)**	**Diluted to final volume (mL)**
Level 1	100	250
Level 2	100	100
Level 3	100	50
Level 4	200	20
Level 5	400	20
Level 6	400	10
Level 7	800	10

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations are maintained.

Table E-10		
Surrogate Recovery Limits		
Surrogate	**Water Recovery Limits**	**Soil Recovery Limits**
Benzo(e)pyrene	78-125	49-129
Terphenyl-d14	72-125	54-126

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Appendix F

Analysis of Phenols Based on Method 8041

109 SCOPE AND APPLICATION

109.1 This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of phenolic compounds by GC/FID. This Appendix is to be applied when SW-846 Method 8041 is requested, and is applicable to extracts derived from any matrices, which are prepared according to the appropriate TestAmerica Pittsburgh sample extraction SOPs. (PITT-OP-0001)

109.2 Table F-1 lists compounds, which are routinely determined by this method and their associated Reporting Limits (RL) for each matrix. RLs given are based on the low-level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

110 SUMMARY OF METHOD

110.1 This method presents conditions for the analysis of prepared extracts for phenols. The phenols are injected onto the GC column and separated and detected by Flame Ionization detection. Quantitation is by the external standard method.

111 DEFINITIONS

111.1 Refer to the LQM for definitions of terms used in this document.

112 INTERFERENCES

112.1 Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

112.2 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the flame ionization detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding sample/reagent contact with plastic materials.

112.3 Interferences extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups have not been determined for this method.

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113 SAFETY

113.1 Refer to section 5 of the Method 8000B SOP for general safety requirements.

114 EQUIPMENT AND SUPPLIES

114.1 Refer to Section 6 of the 8000B section of this SOP. A GC equipped with a flame ionization detector is required.

114.2 Refer to Table F-2 for analytical columns.

114.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

115 REAGENTS AND STANDARDS

115.1 Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

115.2 Refer to Tables F-3 and F-7 for details of calibration standards.

115.3 Surrogate Standards – purchased as a neat material. A 2000 ug/ml solution is made in methanol. See page F10.

Dibromophenol is the surrogate standard. Refer to Table F-4 for details of surrogate standard.

116 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

116.1 Refer to Section 8 of the 8000B section of this SOP.

117 QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP. LCS and MS/MSD control limits are listed in Table F6-A.

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118 PROCEDURE

118.1 Calibration and Standardization

118.2 Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

118.3 Refer to Table F-2 for details of GC operating conditions.

118.4 Initial Calibration

118.5 Refer to Section 10 of the 8000B section of this SOP for details of calibration procedures.

118.6 A six point calibration of each of the individual analyte mixes is generated.

118.7 Refer to Table F-7 for the initial calibration analytical sequence.

118.8 The response for each single-peak analyte will be calculated by the procedures described in the general method for GC analysis.

118.9 Initial Calibration Verification

118.10 The ICV will consist of second source standards of all analytes of interest. Refer to the 8000B section of this SOP for acceptance criteria.

118.11 12 hour Calibration Verification

118.12 The 12 hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration. A mid level calibration standard is used for the 12 hour calibration. Refer to the 8000B section of this SOP for acceptance criteria.

118.13 The retention time windows for any analytes included in the 12 hour calibration are updated.

118.14 Continuing Calibration

118.15 The mid-level calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. If 12 hours elapse analyze the 12 hour standard sequence instead. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

118.16 The daily CCV analysis at a concentration other than the mid level (to meet NELAC requirements) will consist of all of the analytes of interest. All other CCVs will be mid level calibration standards.

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Calibration Controls	Sequence	Control Limit
Calibration Standards	5 pt. Curve (minimum)	≤ 20% RSD – 8041 (alternatively, if the correlation coefficient is >0.99, linear regression may be used).
Second Source	prior to samples	± 20 % of expected value
Retention Time Windows	after calibration, update daily	3 X Standard Deviation
Cont. Cal. Verif. (CCV)	every 20 samples	± 15% D
Note: If the CCV is > ±15% for any one compound, data will be acceptable if the result is J flagged. The result must be less than the RL.		

118.17 Procedure

118.18 Refer to the method 8000B section of this SOP for general procedural requirements.

118.19 Extraction - The extraction procedure is described in SOP No. PITT-OP-0001. Extraction is performed by Method 3520C.

118.20 Suggested gas chromatographic conditions are given in Table F-2.

118.21 Allow extracts to warm to ambient temperature before injection.

118.22 The suggested analytical sequence is given in Table F-6.

119 CALCULATIONS / DATA REDUCTION

119.1 Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

119.2 Surrogate recovery results are calculated and reported for Dibromophenol.

120 METHOD PERFORMANCE

120.1 Current laboratory performance limits are listed in Table F-6. The spiking level should be equivalent to a mid-level calibration.

121 POLLUTION CONTROL

Refer to Section 13 of the 8000B section of this SOP.

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122 WASTE MANAGEMENT

122.1 Refer to Section 14 of the 8000B section of this SOP.

123 REFERENCES

Test Methods for Evaluating Solid Waste, SW-846, Update III, December 1996, Method 8041

124 ATTACHMENTS

124.1 Table F-1: Standard Analyte List and Reporting Limits

124.2 Table F-2: Recommended GC Operating Conditions

124.3 Table F-3: Calibration Levels

124.4 Table F-4: LCS/Matrix Spike and Surrogate Spike Levels

124.5 Table F-5: LCS and Matrix Spike Control Limits

124.6 Table F-6: Suggested Analytical Sequence

124.7 Table F-7: Preparation of Calibration Standards

124.8 Table F-8: Surrogate Recovery Limits

125 REVISION HISTORY

125.1 Revision 12, 09/07/2007

125.2 Changed the format of the SOP to correspond to the new Corporate SOP format.

126 METHOD MODIFICATIONS

126.1 Refer to Section 18 of the 8000B section of this SOP.

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Table F-1		
Method 8041 Reporting Limits		
Compound	**Water RL (ug/L)**	**Soil RL (ug/kg)**
4-Chloro-3-methylphenol	1	33
2,4-Dichlorophenol	1	33
4,6-Dinitro-2-methylphenol	2	67
2,4-Dinitrophenol	2	67
2-Nitrophenol	1	33
4-Nitrophenol	2	67
Pentachlorophenol	2	67
Phenol	1	33
2,3,5,6-Tetrachlorophenol	2	67
2,4,6-Trichlorophenol	2	67

Table F-2	
Instrument Conditions	
Parameter	**Recommended Conditions**
Injection port temp	200°C
Detector temp	250°C
Temperature program	50C for 4 min, 11.0°C/min to 280°C, , 1.1 min hold
Column 1	RTx-200 30m x 0.53mm ID, 1.0µm DF
Column 2	RTx-5 30m x 0.53 mm ID, 0.25µm DF
Injection	2µL
Carrier gas	Helium
Make up gas	Nitrogen

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Table F-3						
Calibration Levels ug/mL						
	Level 1	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**
Phenol	2	10	20	30	40	80
2,4-Dichlorophenol	2	10	20	30	40	80
2-Nitrophenol	2	10	20	30	40	80
4-Chloro-3-Methylphenol	2	10	20	30	40	80
2,4,6-Trichlorophenol	2	10	20	30	40	80
2,3,5,6-Tetrachlorophenol	2	10	20	30	40	80
4-Nitrophenol	2	10	20	30	40	80
Pentachlorophenol	2	10	20	30	40	80
2,4-Dinitrophenol	2	10	20	30	40	80
4,6-Dinitro-2-Methylphenol	2	10	20	30	40	80
Dibromophenol (Surrogate)	2	10	20	30	40	80

Table F-4	
LCS/Matrix Spike and Surrogate Spike levels µg/L	
Compound	**Aqueous**
Phenol	25
2,4-Dichlorophenol	25
2-Nitrophenol	25
4-Chloro-3-Methylphenol	25
2,4,6-Trichlorophenol	25
2,3,5,6-Tetrachlorophenol	25

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Table F-4	
LCS/Matrix Spike and Surrogate Spike levels µg/L	
Compound	**Aqueous**
4-Nitrophenol	25
Pentachlorophenol	25
2,4-Dinitrophenol	25
4,6-Dinitro-2-Methylphenol	25
Dibromophenol (Surrogate)	20

Table F-5

LCS and MS/MSD Control Limits

	Soil Method 8041						Water Method 8041					
	LCS			Spike			LCS			Spike		
Compound	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD	LCL	UCL	RPD
4-Chloro-3-methylphenol	46	122	20	28	135	28	62	123	20	59	117	25
2,4-Dichlorophenol	43	124	20	24	138	29	53	134	20	27	150	20
4,6-Dinitro-2-methylphenol	35	116	20	19	129	26	34	128	20	55	122	34
2,4-Dinitrophenol	33	115	20	16	131	46	45	118	20	50	126	31
2-Nitrophenol	38	128	20	20	126	20	42	143	20	50	126	20
4-Nitrophenol	31	133	20	35	120	44	58	128	20	69	115	25
Pentachlorophenol	56	128	20	24	141	28	50	126	20	54	133	20
Phenol	27	129	20	25	127	28	52	135	20	58	121	27
2,3,5,6-Tetrachlorophenol	48	150	20	28	130	36	68	129	20	31	134	24
2,4,6-Trichlorophenol	42	126	20	38	130	42	54	143	20	53	131	28

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Table F-6	
Suggested Analytical Sequence	
Initial Calibration	
Solvent Blank (optional)	
Calibration Mix	All Levels
ICV (second source standards)	
Up to 20 samples (unless 12 hours comes first)	
Continuing Calibration	Alternate Levels
Up to 20 samples (unless 12 hours comes first)	
Continuing Calibration	Alternate Levels
Repeat	

Calibration Standard Preparation:

Table F-7	
Preparation of Calibration Standards	
Calibration Level	**Stock Standard (uL)**
Level 1	20
Level 2	100
Level 3	200
Level 4	300
Level 5	400
Level 6	800

The Calibration Standards are purchased as certified standards in two separate solutions at 2000 ug/mL for each analyte.

The Standard Mix 1 (Phenols Mixture “A”) includes: 2,3,4,5-Tetrachlorophenol, 2,3,5,6-Tetrachlorophenoland 4,6-Dinitro-2-Methylphenol, in isopropanol.

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The Standard Mix 2 (Phenols Mixture "B") includes: All the compounds in Table F-3 except for 2,3,4,5-Tetrachlorphenol, 2,3,5,6-Tetrachlorophenol and 4,6-Dinitro-2-Methylphenol, in methylene chloride.

The Standard Mix 3 includes the surrogate only, in methanol.

The three mixes are combined into the calibration standards with a final volume of 20mL in methylene chloride using the volumes in Table F-8 for each calibration level.

Table F-8

Surrogate Recovery Limits

Surrogate	Water Recovery Limits	Soil Recovery Limits
2,4-Dibromophenol	31-134	28-115

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Appendix G

Analysis of Herbicides Based on Method 8151A

127 SCOPE AND APPLICATION

This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of Phenoxy Acid Herbicides by GC/ECD. This Appendix is to be applied when SW-846 Method 8151A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate TestAmerica sample extraction SOP (PITT-OP-0001)

Table G-1 lists compounds, which are routinely determined by this method and gives the Reporting Limits (RL) for each matrix. Matrix interferences may result in higher RLs than those listed.

128 SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts for phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by the external standard method.

129 DEFINITIONS

Refer to the LQM for definitions of terms used in this document.

130 INTERFERENCES

130.1 Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

130.2 Chlorinated acids and phenols cause the most direct interference with this method.

130.3 Sulfur may interfere and may be removed by the procedure described in SOP# PITT-OP-0001.

131 SAFETY

131.1 Refer to section 5 of the Method 8000B SOP for general safety requirements.

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132 EQUIPMENT AND SUPPLIES

132.1 Refer to Section 6 of the 8000B section of this SOP. A GC equipped with a Ni_{63} electron capture detector is required.

132.2 Refer to Table G2 for analytical columns.

132.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

133 REAGENTS AND STANDARDS

133.1 Refer to section 7 of the 8000B section of this SOP for general information on reagents and standards.

133.2 Refer to Table G-3 and D-5 for details of calibration and other standards.

134 SAMPLE PREPARATION, PRESERVATION, SHIPMENT AND STORAGE

Refer to section 8 of the 8000B section of this SOP.

135 QUALITY CONTROL

135.1 Refer to Section 9 of the 8000B section of this SOP for quality control requirements.

135.2 Refer to Table G-4 for minimum performance criteria for the initial demonstration of capability.

135.3 Refer to Table G-3 for the components and levels of the LCS and MS mixes.

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136 PROCEDURE

136.1 Calibration and Standardization

136.2 Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

136.3 Initial Calibration

136.4 The calibration stock standard solution is purchased in the acid form and derivatized into the ester form. Refer to Table G-5 for the calibration levels as the acid form, and Tables G-7 and D-8 for the preparation of the calibration standards.

136.5 Refer to Table G-6 for the initial calibration analytical sequence.

136.6 The low level standard must be at or below the laboratory reporting limit. Other standards are chosen to bracket the expected range of concentrations found in samples, without saturating the detector or leading to excessive carryover.

136.7 Refer to Table G-2, for details of GC operating conditions.

136.8 Initial Calibration Verification

136.9 The ICV will consist of a second source standard for all analytes. Refer to the 8000B section of this SOP for acceptance criteria.

136.10 Daily Calibration

136.11 The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

136.12 The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

136.13 Continuing Calibration

136.14 **After every 10 samples a continuing calibration is analyzed.** The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

136.15 A daily CCV analysis at a concentration other than the mid level (to meet NELAC requirements) will consist of all analytes of interest.

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Calibration Controls	Sequence	Control Limit
Calibration Standards	5-pt. (min) linearity	≤20% RSD (alternatively, if the correlation coefficient is >0.99, linear regression may be used).
Cont. Cal. Verif. (CCV)	prior to and after every 10 injections	±15% D
Second source verification	prior to samples	± 20 % of expected value
Retention Time Windows (RTW)	After calibration, update daily	± 3x SD
Note: If the CCV is > ±15% for any one compound, data will be acceptable if the result is J flagged. The result must be less than the RL.		

136.16 Procedure

136.17 Refer to the method 8000B section of this SOP for procedural requirements.

136.18 Extraction

The extraction procedure is described in SOP #PITT-OP-0001.

136.19 Cleanup

The alkaline hydrolysis and subsequent extraction of the basic solution described in the extraction procedure (SOP PITT-OP-0001) provides an effective cleanup.

136.20 Analytical Sequence

Refer to Table G-6 for the suggested analytical sequence.

136.21 Gas Chromatography

Chromatographic conditions are listed in Table G-2.

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137 CALCULATIONS / DATA REDUCTION

137.1 Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

138 METHOD PERFORMANCE

138.1 Multiple laboratory performance data has not been published by the EPA for this method. Table G-4 lists minimum performance standards required by TestAmerica for the four replicate initial demonstration or capability (required by Section 12.2 of the 8000B part of this SOP) for this method. The spiking level should be equivalent to a mid level calibration.

139 POLLUTION CONTROL

Refer to Section 13 of the 8000B section of this SOP.

140 WASTE MANAGEMENT

Refer to Section 14 of the 8000B section of this SOP.

141 REFERENCES

141.1 Test Methods for Evaluating Solid Waste, SW-846, Update III, December 1996, Method 8151A

141.2 SOP # PITT-QA-DoD-0001, Implementation of DoD QSM Version 3 January 2006, current version.

142 ATTACHMENTS

142.1 Table G-1: Standard Analyte List and Reporting Limits

142.2 Table G-2: Recommended GC Operating Conditions

142.3 Table G-3: LCS/Matrix Spike and Surrogate Spike Levels

142.4 Table G-4: LCS and Matrix Spike Control Limits

142.5 Table G-5: Calibration Levels

142.6 Table G-6: Suggested Analytical Sequence

142.7 Table G-7: Stock and Intermediate Standard Concentrations as Free Acids

142.8 Table G-8: Preparation of Calibration Standards

142.9 Table G-9: Surrogate Recovery Limits

143 REVISION HISTORY

143.1 Revision 12, 09/07/2007

143.2 Changed the format of the SOP to correspond to the new Corporate SOP format.

144 METHOD MODIFICATIONS

144.1 Refer to Section 18 of the 8000B section of this SOP.

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Table G-1

Standard Analyte list

Compound	CAS Number	Reporting Limit as free acid (µg/L or µg/kg)			
		Aqueous	Soil	Waste	TCLP
2,4-D	94-75-7	4	80	4000	500
2,4-DB	94-82-6	4	80	4000	---
2,4,5-TP (Silvex)	93-72-1	1	20	1000	500
2,4,5-T	93-76-5	1	20	1000	---
Pentachlorophenol	87-86-5	1	10	500	---
Dalapon	75-99-0	2	40	2000	---
Dicamba	1918-00-9	2	40	2000	---
Dichloroprop	120-36-5	4	80	4000	---
Dinoseb	88-85-7	0.6	12	600	---
MCPA	94-74-6	400	8000	400,000	---
MCPP	93-65-2	400	8000	400,000	---

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.	Dilution Factor
Groundwater	1000 mL	10 mL	20
Low-Level Soil without GPC	50 g	10 mL	20
High-Level Soil/Waste	1 g	10 mL	20
TCLP Leachates	100 mL	10 mL	20

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 20 times lower than those in Table G-1.

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Table G-2	
Instrumental Conditions	
PARAMETER	**Recommended conditions**
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	DB-5MS or RTX 5, 30m x 0.32mm id, 0.5um
Column 2	DB-1701 or Rtx-1701, 30m x 0.53mm id, 1.0um
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Recommended conditions should result in resolution of all analytes listed in Table G-1.

Table G-3				
LCS/Matrix Spike and Surrogate Spike levels (µg/L or µg/kg[1])				
	Aqueous	**Soil**	**Waste**	**TCLP**
2,4-D	16	320	16000	160
2,4,5-TP (Silvex)	4	80	4000	40
2,4,5-T	4	80	4000	40
2,4-DB	16	320	16000	---
Dalapon	8	160	8000	---
Dicamba	8	160	8000	---
Dichloroprop	16	320	16000	---
Dinoseb	2.4	48	2400	---
MCPA	1600	32000	1600000	---

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Table G-3				
LCS/Matrix Spike and Surrogate Spike levels (µg/L or µg/kg[1])				
MCPP	1600	32000	1600000	---
Pentachlorophenol	2	40	2000	---
DCAA (surrogate)	10	200	10000	100

[1] LCS, MS and Surrogate spikes are as the free acid.

Table G-4 LCS and Matrix Spike Control Limits

	Water LCS				**Water Matrix Spike**			
Compound	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
2,4-D	16	65	126	32	16	36	113	52
Dalapon	8	65	112	35	8	32	131	60
2,4-DB	16	71	134	29	16	53	118	40
Dicamba	8	77	122	24	8	10	150	42
Dichlorprop	16	80	129	20	16	51	102	30
Dinoseb	2.4	22	127	70	2.4	15	161	79
MCPA	1600	75	129	35	1600	14	139	44
MCPP	1600	67	143	35	1600	18	145	34
Pentachlorophenol	4	67	139	22	4	54	126	34
2,4,5-TP (Silvex)	4	70	122	34	4	48	99	42
2,4,5-T	4	62	130	36	4	33	105	63
DCAA	10	53	119	20	10	53	119	20
	Soil LCS				**Soil Matrix Spike**			
Compound	AMT	LCL	UCL	RPD	AMT	LCL	UCL	RPD
2,4-D	320	61	120	27	320	10	111	107
Dalapon	160	61	110	37	160	10	168	108
2,4-DB	320	61	128	29	320	10	121	122
Dicamba	160	70	116	23	160	14	132	105
Dichlorprop	320	67	127	14	320	10	113	118
Dinoseb	48	15	122	61	48	10	207	188
MCPA	32000	60	129	19	32000	10	98	154
MCPP	32000	64	135	22	32000	12	115	99

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Table G-4 LCS and Matrix Spike Control Limits								
Pentachlorophenol	40	75	122	16	40	34	155	65
2,4,5-TP (Silvex)	80	67	116	26	80	10	120	98
2,4,5-T	80	57	126	35	80	10	108	164
DCAA	200	42	125	20	200	42	125	20

Table G-5					
Calibration Levels					
Compound	**Concentration levels in ug/ml (as free acids)**				
2,4-D	0.0200	0.0401	0.0802	0.1600	0.3210
DCAA (surrogate)	0.0201	0.0402	0.0803	0.1610	0.3210
2,4-DB	0.0200	0.0400	0.0800	0.1600	0.3200
2,4,5-TP (Silvex)	0.0050	0.0100	0.0200	0.0401	0.0802
2,4,5-T	0.0050	0.0100	0.0200	0.0401	0.0802
Pentachlorophenol	0.0025	0.005	0.0100	0.0200	0.0400
Dalapon	0.01	0.02	0.0401	0.0802	0.1600
Dicamba	0.0100	0.0199	0.0398	0.0797	0.1590
Dichloroprop	0.0200	0.0400	0.0801	0.1600	0.3200
MCPP	2.001	4.0020	8.0030	16.0060	32.0130
Dinoseb	0.0030	0.0060	0.0120	0.0240	0.0480
MCPA	2.0000	4.0000	8.0000	16.0000	32.0000

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Table G-6	
Suggested Analytical Sequence	
Initial Calibration	
Solvent Blank (optional)	
Individual Mix (all analytes)	All Levels
ICV (all analytes)	
Solvent Blank	
Up to 20 samples (unless 12 hours comes first)	
Solvent blank (optional)	
CCV (all analytes)	Mid-Level
Up to 20 samples (unless 12 hours comes first)	
CCV (all samples)	Mid-Level, etc.

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Table G-7		
Stock and Intermediate Standard Concentrations as Free Acids (ug/mL)		
Compound	**Stock Standard**	**Intermediate Standard**
2,4-D	200	20
2,4-DB	200	20
2,4,5-TP (Silvex)	50	5
2,4,5-T	50	5
Pentachlorophenol	25	2.5
Dalapon	100	10
Dicamba	100	10
Dichloroprop	200	20
Dinoseb	30	3
MCPA	20000	2000
MCPP	20000	2000
DCAA (surrogate)	200	20

The stock standard is a certified solution in hexane of methyl esters, however, the certified concentrations are based on the free acid. The intermediate standard is prepared by dilution 1.0 mL of the stock standard to a 10 ml final volume in hexane.

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Table G-8	
Preparation of Calibration Standards	
Calibration Level	**Intermediate Std. (uL)**
Level 1	40
Level 2	80
Level 3	400
Level 4	320
Level 5	640

The working standards are prepared by diluting the volumes of the intermediate standard noted in TableG-8 to a 40.0 mL final volume in hexane except for the Level 3 standard, which is taken to a 100 mL final volume in hexane.

Table G-9		
Surrogate Recovery Limits		
Surrogate	**Water Recovery Limits**	**Soil Recovery Limits**
DCAA	53-119	42-125

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Appendix H

DOD QSM Version 3 Requirements GC/HPLC

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table H-1

Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/HPLC)

QC Check	Definition	Purpose	Evaluation
Breakdown Check 8081A: Endrin, DDT 8270C: DDT	Analysis of s standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degradation of either DDT or Endrin exceeds method–specified criteria, corrective action must be taken before proceeding with calibration
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified	To verify the identification of an analyte	This is a required QC procedure. All positive results must be confirmed.
CCV	This verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models	To verify that instrument response is reliable, and has not changed significantly from the current ICAL	If the values for the analytes are outside the acceptance criteria, the initial calibration may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging
Demonstrate Acceptable Analyst Capability	Analyst runs QC samples in series to establish his/her ability to produce data of acceptable accuracy and precision	To establish the analysts' ability to produce data of acceptable accuracy and precision	The average recovery and standard deviation of the replicate must be within designated acceptance criteria.
Duplicate Sample	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are taken and prepared and analyzed identically.	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix	To provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the sample and the duplicate

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Table H-1

Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/HPLC)

QC Check	Definition	Purpose	Evaluation
ICAL	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method	To establish a calibration curve for the quantification of the analytes of interest	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Internal Standards	A known amount of standard added to all standards and samples as a reference for evaluating and controlling the precision and bias of the applied analytical method	To verify that the analytical system is in control	Any sample associated with out-of-control results must be reanalyzed.
LCS containing all analytes required to be reported	A QC standard of known composition prepared using reagent free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern.	To evaluate method performance by assessing the ability of the lab/analyst to successfully recover the target analytes from a control (clean) matrix.	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the accuracy/bias of the measurement system.
MS	A sample prepared by adding a know amount of targeted analyte(s) to an aliquot of a specific environmental sample	To assess the performance of the method as applied to a particular matrix	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty
MSD	A 2nd replicate MS prepared in the lab, spiked with an identical	To assess the performance of the	When compared to the MS, the MSD will provide

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Table H-1

Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/HPLC)

QC Check	Definition	Purpose	Evaluation
	the lab, spiked with an identical, known amount of targeted analyte(s), and analyzed to obtain a measure of the precision of the recovery for each analyte	performance of the method as applied to a particular matrix and provide information of the homogeneity of the matrix.	MS, the MSD will provide information on the heterogeneity of the sample matrix.
MDL Verification Check	A low-level spike taken through the prep and analytical steps at approximately 2x the MDL used to verify that the laboratory can detect analytes at the calculated MDL	To validate the MDL on an ongoing basis	If the MDL verification check fails, reprep/reanalyze at a higher level to set a higher MDL or the MDL study must be repeated.
MB	A sample of a matrix similar to the batch of associated samples in which no target analytes or interferences are present at concentrations that impact the analytical results.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data.	This QC is used to measure lab accuracy/bias. The MB could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or B-Flag results for all samples in prep batch. For common lab contaminants, no analytes detected > RL. See DoD Box D-5; & Sec. D.1.1.1
MDL Study	The process to determine the minimum concentration of an analyte 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.	To determine the lowest concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.	MDLs must be established prior to sample analysis. The RL or LOQ is at least 3x the MDL. Used in combination with the MDL verification check to validate the MDL on an ongoing basis.
RT window position establishment for each analyte (chromatographic	Determination of the placement of the RT window (start/stop time) of each analyte or group of analytes as it elutes through the	To identify analytes of interest	Incorrect window position may result in false negatives, require additional manual

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Table H-1

Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/HPLC)

QC Check	Definition	Purpose	Evaluation
methods only)	chromatographic column so that analyte identification can be made during sample analysis. This is done during the initial calibration		integrations, and/or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
RT window verification for each analyte (chromatographic methods only)	A standard is used to verify that the width and position of the RT windows are valid so that accurate analyte identification can be made during sample analysis	To minimize the occurrence of both false positive and false negative results at each calibration verification.	The peaks from the standard used are compared to the RT window established during the ICAL to verify that the analytes of interest still fall within the window.
RT window width calculate for each analyte and surrogate (non-MS chromatographic methods only)	Determine the length of time between the sample injection and the appearance of a peak at the detector. The total length of time (window) is established for each analyte or groups of analytes and is set for complete elution of analyte peaks. It is based upon a series of analyses and statistical calculations that establish the measured band on the chromatogram that can be associated with a specific analyte or group of analytes.	To ensure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the standards and sample matrix to be analyzed. It is done to minimize the occurrence of both false positive and false negative results	Used to evaluate continued system performance. Tight RT windows may result in false negatives and/or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide RT windows may result in false positive results that cannot be confirmed upon further analysis.
Second source calibration verification	A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration.	To verify the accuracy of the initial calibration	The concentration of the 2nd source calibration verification, determined from the analysis, is compared to the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.
Surrogate spike (organic analysis only)	A pure substance with properties that mimic the analyte of interest. Surrogates are compounds unlikely to be found in	To assess the ability of the method to successfully recover specific non-target	Whereas the MS is normally done on a batch-specific basis, the surrogate spike is done on

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Table H-1

Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/HPLC)

QC Check	Definition	Purpose	Evaluation
	environmental samples to evaluate analytical efficiency by measuring their % Recovery.	analytes from an actual matrix.	a sample-specific basis. Taken with the information derived from other spikes (LCS; MS), the bias in the analytical system can be determined.

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.
3. If the requirements in the DoD tables do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

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DoD QSM Version 3:

Appendix DOD-B Quality Control Requirements Summary

Table H-2

Organic Analysis by GC and HPLC – Methods 8081, 8082, 8141, 8151, and 8310

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria*
IDOC	Per Instrument/Analyst	DoD acceptance criteria if available; otherwise method specific criteria	Correct / Repeat for those analytes which failed criteria	NA
MDL	Annually or quarterly MDL Checks performed	40 CFR 136B; MDL verification checks must produce a signal at least 3x the instrument's noise level.	Run MDL check at higher level and set MDL higher or reconduct MDL study	NA
RT window	At method set-up and after major instrument maintenance	RT width is $\pm$ 3x standard deviation for each analyte RT from 72-hour study	NA	NA
Breakdown check (Endrin/DDT-8081 only)	Daily prior to analysis of samples	Degradation $\leq$ 15% for both Endrin & DDT	Correct problem then repeat breakdown check	NA
ICAL	Initial 5-point calibration prior to sample analysis	One of the options below (Except for 8082. which may only use Option 1 or 2) **Option 1:** RSD for each analyte $\leq$ 20% **Option 2:** linear least squares regression: r $\geq$ 0.995 **Option 3:** non-linear regression: Coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for 2nd order, 7 points shall be used for 3rd order)	Correct problem then repeat initial calibration	NA
2nd Source calibration verification	Once after each initial calibration	Value of 2nd source for all analytes within $\pm$ 20% of expected value	Correct problem and verify 2nd source standard. Rerun, if that fails, correct problem and repeat initial calibration.	NA

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Table H-2

Organic Analysis by GC and HPLC – Methods 8081, 8082, 8141, 8151, and 8310

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria*
RT window position establishment for each analyte	Once per ICAL and at the beginning of the analytical shift	Position shall be set using midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift	NA	NA
RT window verification for each analyte & surrogate	Each calibration verification standard	Analyte within established window	Correct problem, then reanalyze all samples analyzed since the last acceptable RT check. If they fail, repeat ICAl and reset RT window	ICAL – NA CCV – apply Q-flag to all results for analytes outside of RT window
Calibration verification (ICV) and continuing (CCV)	ICV – Daily, before sample analysis CCV – After every 10 field samples and at end of analysis sequence. (DoD Box 58: CCV standards shall be at or below the middle of the calibration range)	All analytes within +/- 20% of expected value from ICAL (Data associated with an unacceptable CCV may be fully usable under the following conditions: CCV (high bias) and samples ND, then raw data may be reported with appropriate flag 2. CCV (low bias) and samples exceed maximum regulatory limit/decision level (DoD Box 60: Project specific permission from appropriate DoD personnel is required to report data generated from a run with noncompliant CCV.)	ICV – Correct problem, rerun ICV, if fails, repeat ICAL CCV – Correct problem, repeat CCV and reanalyze all samples since the last good CCV (DoD Box 59...if the lab chooses to demonstrate the success of routine corrective action through the use of 2 consecutive CCVs, then the concentrations of the two CCVs must be a two different levels within the original calibration curve with at least one falling below the middle of the calibration range.)	ICV – NA CCV – apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV, if reanalysis is not possible
MB	One per prep batch	No analytes detected > ½ RL For common lab contaminants, no analytes ≥ RL	Correct problem, then see criteria in box D-5; if required, reprep/reanalyze MB and all associated samples	Apply B-flag to all results for the contaminated analyte for all samples in the associated prep batch
MS	One per prep batch per matrix	For matrix evaluation, use DoD specified QC criteria for LCS	Examine the project-specific DQOs. Contact client for additional	Apply J-flag to specific analyte(s) in the parent

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Table H-2

Organic Analysis by GC and HPLC – Methods 8081, 8082, 8141, 8151, and 8310

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria*
		LCS	client for additional corrective action measures.	in the parent sample
MSD or Sample Duplicate	One per prep batch per matrix	RPD ≤ 30% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact client for additional corrective action measures.	Apply J-flag to specific analyte(s) in the parent sample
Surrogate	All field and QC samples	DoD specified QC criteria if available, otherwise method specific criteria or lab's own in-house criteria	For QC and field samples, correct problem, reprep/reanalyze all failed samples in the associated prep batch if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary	Apply J-flag for specific analyte(s) in all field samples collected from the same site matrix as the parent. Apply Q-flag to QC samples for specific analyte(s)
Confirmation of Positive Results	All positive results must be confirmed (exceptions: 8081A, toxaphene & technical chlordane 8015B, GRO, DRO	Calibration and QC criteria same for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%	NA Note: Report the higher of the two confirmed results unless overlapping peaks are causing erroneously high results – document in case narrative	Apply J-flag if RPD > 40% or Q-flag if sample is not confirmed. Discuss in the case narrative
Results reported between LOD and LOQ			Apply J-flag to all results between LOD (MDL) and LOQ (RL)	
Manual Integration	When manual integrations are performed	Raw data shall include a complete audit trail for those manipulations, raw data output showing the results of the MI (i.e., chromatograms of manually integrated peaks), and notation of		Apply M-flag to MI data

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Table H-2

Organic Analysis by GC and HPLC – Methods 8081, 8082, 8141, 8151, and 8310

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria*
		rationale, date, and signature/initials of person performing manual operation.		

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.
2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.
3. If the requirements in the DoD tables do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.
4. * TestAmerica Pittsburgh does not use all the flags as indicated in the QSM.

Title: Acid Digestion of Soils

Method: SW846 Method 3050B

Approvals (Signature/Date):

William Reinheimer	10/5/07 Date	Larry Matko	9/19/07 Date
Technical Manager		Acting Health & Safety Manager	
Nasreen DeRubeis	9/19/07 Date	Larry Matko	9/19/07 Date
Quality Assurance Manager		Laboratory Director	

This SOP was previously identified as SOP No. PITT-IP-0002, Rev.6.

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of soil samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) as specified in SW846 Method 3050B.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP or ICP/MS for the elements listed in Table I (Appendix A). Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 12.0 of this SOP are met.

1.3. This method is not a total digestion, but will dissolve almost all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludges, wastes, sediments, wipes and tissues.

2. SUMMARY OF METHOD

A representative 1-gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP analysis. The digestates are then filtered and diluted to 100 mL/100 g.

3. DEFINITIONS

Additional definitions of terms used in this SOP may be found in the glossary of the QMP.

3.1. Total Metals: The concentration determined on an unfiltered sample following digestion. Note that this method is designed to determine the total *environmentally available* metals.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

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4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix C for additional contamination control guidelines.

4.3. Boron and silica from the glassware will grow into the sample solution during and following sample processing. For critical low-level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric media.

4.7. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. All heating of samples must be carried out in a fume hood.

5.4. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.5. 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

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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.
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.
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.			

5.6. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.7. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit or under other means of mechanical ventilation.

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5.8. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, hot block, or other heating source capable of maintaining a temperature of 90-95°C.

6.2. Thermometer that covers a temperature range of 0-150°C.

6.3. Hot block Disposable Digestion Cup (from Environmental Express).

6.4. Vapor recovery device (Watch glasses, ribbed or other device).

6.5. Whatman No. 41 filter paper or equivalent.

6.6. Funnels or equivalent filtration apparatus.

6.7. Centrifugation equipment (if desired method of removing particulates is centrifugation).

6.8. Graduated cylinder or equivalent capable of measuring 100 mL within 3% accuracy.

6.9. Analytical balance capable of accurately weighing to the nearest 0.01 grams.

6.10. Repipetors or suitable reagent dispensers.

6.11. Calibrated automatic pipettes with corresponding pipet tips or Class A glass volumetric pipettes.

6.12. Class A volumetric flasks.

6.13. pH indicator strips (pH range 0 - 6).

6.14. Plastic bottles.

6.15. Teflon chips.

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7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom TestAmerica solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working ICP LCS/MS spike solution: The ICP LCS/MS working spike solution is provided directly by the vendor, no further standard preparation is necessary.

7.4. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom TestAmerica solution, a solution must be purchased from a designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.5. Laboratory Control Samples (LCSs) and matrix spike samples are prepared as described in Sections 9.5 and 9.6. Refer to Table II (Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for the ICP LCS and matrix spike preparations.

7.6. Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.7. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.8. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.9. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.10. 30% Hydrogen peroxide (H_2O_2), reagent or ultrex grade.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Soil and wipe samples do not require preservation but must be stored at 4 °C ± 2 °C until the time of analysis. Tissue samples will be stored frozen.

9. QUALITY CONTROL

Table III (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 3050B the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest that have been carried through the entire analytical procedure. MDL's must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in TestAmerica QA Policy PITT-QA-007. The spike level should be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the TestAmerica reporting limit.

9.1.2. Initial Demonstration Study- This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

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9.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOPs (PITT-MT-0001 and PITT-MT-0020).

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of Teflon chips, to which all reagents specific to the method are added and then carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOP's. If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be redigested.

9.4.1. Soil method blanks are prepared by taking 1 g of Teflon chips through the procedure described in Section 10.10.

9.5. Laboratory Control Sample (LCS) - One LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOP's. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Table II provides the details regarding the stock, working standards and final spike concentrations for ICP. Refer to Section 7.3 for instructions on preparation of the LCS.

9.5.1. The LCS is prepared by spiking a 1 g aliquot of Teflon chips with 1 mL of the working LCS/MS spike solution (7.3). The LCS is then processed as described in Section 10.10.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred. Table II provides the details regarding the stock, working standards and final matrix spike concentrations for ICP. Refer to Section 7.3 for instructions on preparation of the working matrix spike solutions.

9.6.1. The soil matrix spike sample is prepared by spiking a 1 g aliquot of a sample with 1 mL of the working LCS/MS spike solution (7.3). The matrix spike sample is then processed as described in Section 10.10.

9.7. Quality Assurance Summaries - Certain clients may require specific project or program QC, which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. PROCEDURE

10.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

10.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.3. Hotplate or hot block temperature must be verified daily for each unit used and must be recorded on either the metals preparation log or in a hotplate/hotblock temperature logbook. The hotplate/hotblock temperature should be verified by measuring the temperature of a beaker of reagent water placed on each hotplate/hotblock. For block digestors, use a tube containing water.

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10.4. All preparation procedures must be carried out in a properly functioning hood.

10.5. All samples are to be checked out of sample control with the chain of custody documentation filled out completely.

10.6. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample. The use of automatic label printing programs is recommended to reduce transcription errors (Quantims option).

10.7. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment etc.) contact the lab supervisor or project administrator for further instructions. In some cases it may be more appropriate to process these samples as solids.

10.8. If possible prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

10.9. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

10.10. Preparation of Soils, Sediments, Sludges,Tissues or Wipes for Analysis by ICP or ICP/MS.

10.10.1. Mix sample thoroughly by stirring with a clean plastic or wooden spoon or spatula.

10.10.1.1. Refer to PITT-QA-0024 for subsampling procedures.

10.10.1.2. Mixing is not required for wipe samples because the entire wipe is used in the digestion.

10.10.2. For each sample, weigh a 1.0 gram portion of solid and record the exact weight to the nearest 0.01 g. A 2 gram sample size may also be used if needed to meet the reporting limits.

10.10.2.1. For wipe samples, the entire sample is digested and no subsampling is required.

10.10.3. Measure additional aliquots of the designated samples for the MS and MSD analyses.

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10.10.3.1. Due to the unique nature of wipe samples, an MS and MSD are not practical. An LCS/LCSD is used to satisfy precision and accuracy requirements for wipes.

10.10.4. Spike each of the MS and MSD aliquots with 1 mL of the working LCS/MS spiking solution (7.3).

10.10.5. Measure 1 g of Teflon chips into a beaker for the method blank.

10.10.5.1. Measure 0.5 mL of reagent water into a beaker for the method blank for wipe samples.

10.10.6. Measure 1 g of Teflon chips into a beaker for the LCS. Spike the LCS aliquot with 1 mL of the working LCS/MS spiking solution (7.3).

10.10.6.1. Measure 0.5 mL of reagent water into a beaker for the LCS and LCSD for wipe samples. Spike the LCS and LCSD aliquots with 0.5 mL of the working LCS/MS spiking solution (7.3).

10.10.7. Add 10 mL of 1:1 HNO_3 and mix the sample.

10.10.7.1. For wipes, add 5 mL of 1:1 HNO_3 and mix the sample.

10.10.8. Cover with a ribbed watch glass.

10.10.9. Heat sample to 95°C and reflux for 10 minutes without boiling, using a vapor recovery device.

Note: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be reprepared.

10.10.10. Allow sample to cool.

10.10.11. Add 5 mL of concentrated HNO_3 and replace vapor recovery device.

10.10.11.1. For wipes, add 2.5 mL of concentrated HNO_3 and replace vapor recovery device.

10.10.12. Reflux at 95°C for 30 minutes. (Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.)

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10.10.13. If brown fumes are observed, repeat steps 10.10.11 and 10.10.12 until no more fumes are evolved.

10.10.14. Using a vapor recovery device, allow the sample to evaporate to 5 - 10 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry. Alternatively heat at 95°C for 2 hours.

10.10.15. Allow the samples to cool.

10.10.16. Add 2 mL of reagent water and 3 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

10.10.16.1. For wipes, add 1 mL of reagent water and 1.5 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

10.10.17. Replace the vapor recovery device and heat sample until effervescence subsides.

10.10.18. Allow the sample to cool.

10.10.19. Continue adding 30% H_2O_2 in 2 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged.

10.10.19.1. For wipes, continue adding 30% H_2O_2 in 1 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged.

Note: Do not add more than a total of 10 mL (5 ml for wipe samples) of 30 % H_2O_2.

10.10.20. Continue heating at 95°C until the volume is reduced to approximately 5 mL. Alternatively the sample may be heated for 2 hours.

10.10.21. Add 10 mL of concentrated HCl and reflux for an additional 15 minutes without boiling.

10.10.21.1. For wipes, add 5 mL of concentrated HCl and reflux for an additional 15 minutes without boiling.

Note: Antimony and silver have poor solubility in dilute nitric acid solution. Therefore it is strongly recommended that these elements are determined by the ICP procedure that includes HCl as the final digestion acid.

10.10.22. Allow the sample to cool.

10.10.23. Wash down beaker walls and vapor recovery device with reagent water.

10.10.24. Filter sample through Whatman 41 filter paper or equivalent into a pre-weighed bottle. Other measuring bottles (for example, Corning Snap Seals™) may be used if their average error is documented and is better than ± 2%. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material

10.10.25. Dilute sample to 100 mL or 100g with reagent water. The sample is now ready for analysis.

10.10.25.1. For wipe samples, dilute sample to 50 mL or 50g with reagent water. The sample is now ready for analysis.

Note: This SOP allows for samples to be weighed instead of measured volumetrically. This assumes the density of the diluted sample is close to 1.0 g/mL (See Section 18.1.2).

10.11. Documentation and Record Management

10.11.1. The preparation benchsheet should, at a minimum, include the following information:

- Preparation date, analyst name, matrix, prep type (ICP or ICP/MS), SOP reference.
- Sample ID, initial weight/volume and final weight/volume.
- Standards Documentation (source, lot, prep date, volume added).
- Analyst Signature.
- Reviewer's Signature and date.

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11. CALCULATIONS / DATA REDUCTION

Not Applicable.

12. METHOD PERFORMANCE

12.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. In general, the matrix spike recovery should fall within +/- 25% and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in the determinative SOPs. The laboratory control samples should recover within 20% of the true value until in house control limits are established. Acceptance criteria are given in the determinative SOPs. **Refer to PITT-QA-DoD-0001 for specific DoD QC requirements.**

12.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

12.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13. POLLUTION CONTROL

13.1. 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.

13.2. This method does not contain any specific modifications that serve to minimize or prevent pollution.

14. WASTE MANAGEMENT

14.1. The following waste streams are produced when this method is carried out.

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14.1.1. Contaminated disposable materials utilized for the analysis. These items are placed in trash containers which are emptied in the general trash dumpster located near the shipping/receiving dock.

15. REFERENCES

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996. Method 3050B.

15.2. PITT-MT-0001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010B and Method 200.7.

15.3. PITT-MT-0020, Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry (ICPMS) for Methods 200.8, 6020 & ILM05.2.

15.4. QA-003, TestAmerica QC Program.

15.5. QA-004, Rounding and Significant Figures.

15.6. PITT-QA-007, Method Detection Limits.

15.7. PITT-QA-0024, Subsampling.

15.8. PITT-QA-DoD-0001, Implementation of the DoD QSM Version 3.

16. ATTACHMENTS

16.1. Figure 1 – Soil Sample Preparation

16.2. Figure 2 – Soil Sample Preparation (Continued)

16.3. Appendix A – Tables

16.3.1. Table I – Method 3050B Approved Analyte List

16.3.2. Table II – ICP & ICPMS Soil Matrix Spike and LCS Levels

16.3.3. Table III – Summary of Quality Control Requirements

16.4. Appendix B – Metals Preparation Bench Sheet

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16.5. Appendix C – Contamination Control Guidelines

17. REVISION HISTORY

17.1. Revision 7, 09/12/07

17.1.1. Changed laboratory name to TestAmerica.

17.1.2. Changed the format of the SOP to correspond to the new Corporate SOP format.

17.1.3. Added the requirement to use a solid matrix for the method blanks and Laboratory Control Samples.

18. METHOD MODIFICATIONS

18.1. Modifications/Interpretations from reference method.

18.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants, as defined in the determinative SOPs, are allowed up to two times the reporting limit in the blank following consultation with the client. **Refer to PITT-QA-DoD-0001 for specific DoD QC requirements.**

18.1.2. This SOP allows for aqueous samples to be weighed instead of measured volumetrically. This assumes the density of the sample is close to 1.0 g/mL. Samples with large amounts of sediment or suspended solids, sludges, non-aqueous liquids must be processed volumetrically. Weighing samples directly into the digestion vessel minimizes the potential for cross contamination, offers improved accuracy over the use of graduated cylinders (comparable to volumetric flask accuracy), uses less glassware and is more efficient.

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Figure 1. Soil Sample Preparation (Section 10.10)

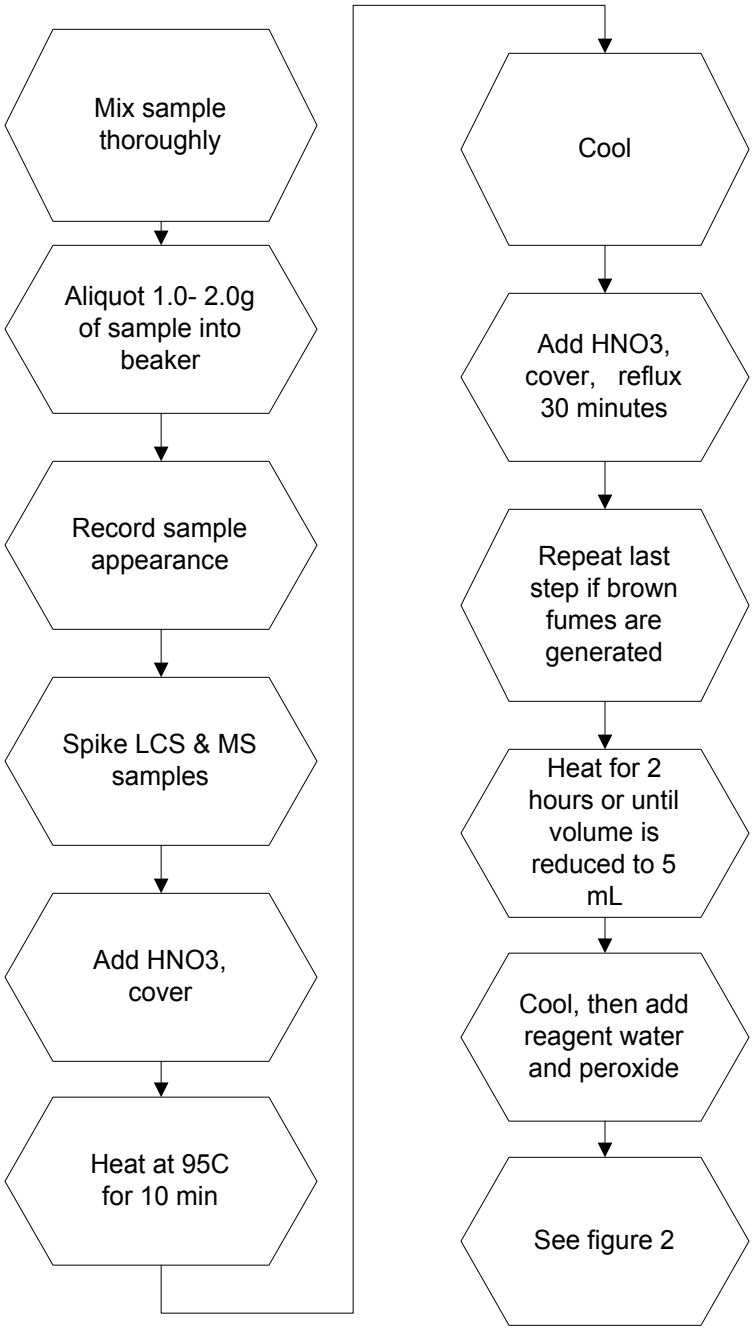

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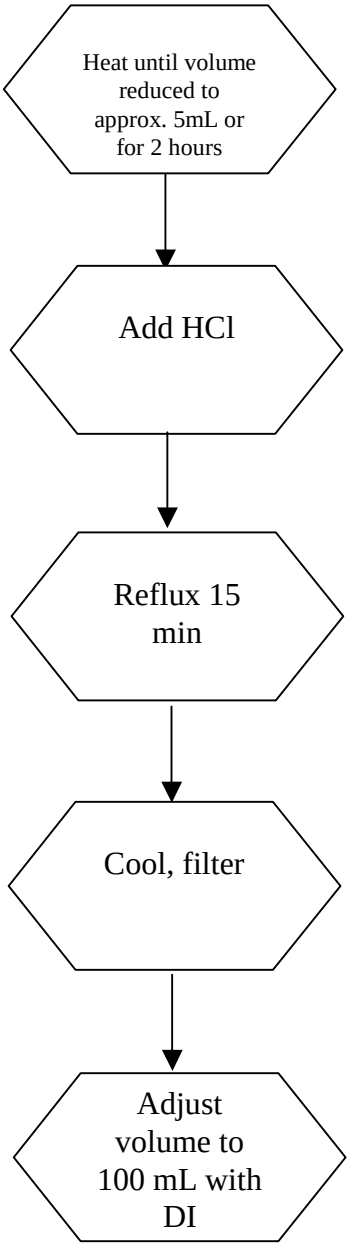
Figure 2. Soil Sample Preparation (continued)



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APPENDIX A

TABLES

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TABLE I. Method 3050B Approved Analyte List

ELEMENT	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

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TABLE II. ICP & ICPMS Soil Matrix Spike and LCS Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/MS Level* (ug/L)	Soil LCS/MS Level ** (mg/Kg)
Aluminum	200	2000	200
Antimony	50	500	50
Arsenic	200 (ICP); 4 (ICPMS)	2000 (ICP); 40 (ICPMS)	200 (ICP); 4 (ICPMS)
Barium	200	2000	200
Beryllium	5	50	5
Cadmium	5	50	5
Calcium	5000	50000	5000
Chromium	20	200	20
Cobalt	50	500	50
Copper	25	250	25
Iron	100	1000	100
Lead	50 (ICP); 2 (ICPMS)	500 (ICP); 20 (ICPMS)	50 (ICP); 2 (ICPMS)
Lithium	100	1000	100
Magnesium	5000	50000	5000
Manganese	50	500	50
Molybdenum	100	1000	100
Nickel	50	500	50
Phosphorous	1000	10000	1000
Potassium	5000	50000	5000
Selenium	200 (ICP); 1 (ICPMS)	2000 (ICP); 10 (ICPMS)	200 (ICP); 1 (ICPMS)
Silver	5	50	5
Sodium	5000	50000	5000
Strontium	100	1000	100
Thallium	200 (ICP); 5 (ICPMS)	2000 (ICP); 50 (ICPMS)	200 (ICP); 5 (ICPMS)
Vanadium	50	500	50
Zinc	50	500	50
Boron	100	1000	100
Silica	1000	10000	1000
Tin	200	2000	200
Titanium	100	1000	100

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* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.3) to 100 mL of sample.

** Final soil spike concentration based on the addition of 1.0 mL working spike (7.3) to 1.0 g of sample (or 1.0 g of Teflon chips for the LCS)/100 mL final volume (assumes 100% solids).

TABLE III. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA[1]	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: PITT-MT-0001 & PITT-MT-0020	Redigest and reanalyze samples.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: PITT-MT-0001 & PITT-MT-0020	Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: PITT-MT-0001 & PITT-MT-0020	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: PITT-MT-0001 & PITT-MT-0020	See Corrective Action for Matrix Spike.

[1] **For specific DoD requirements, refer to PITT-QA-DoD-0001.**

APPENDIX B - TESTAMERICA METALS PREPARATION BENCHSHEET

TestAmerica Pittsburgh ***Metals Preparation Log*** Batch No. 7232156 Page 1 of 1

v1.0.5 - Last Revised 12/30/2005

Method 3050B	Matrix Solid	Start Time 0945	SDG -
Analyst example	Date 082007	Lot Numbers	
MS STLPA-MS-A, STLPA-MS-B, STLPA-MS-C		C7H030157	
Lab Lot # C7H030157			
Analyst Sign:			

Sample ID	Initial Wt/Vol g/mL	Final Vol mL	Comments
J3500			
J3501			
J3503			
J3505			
J3507			
J3509			
J350A			
J350C			
J350F			
J350H			
J350K			
J350N			
J350R			
J350W			
J351D			
J35X4			
J35X6			
J35X8			
J35X9			
J35XM			
J35XM S			
J35XM D			
J47LA B			
J47LA C			

SAMPLE CODING: B-Blank C-Check L-Check Duplicate S-Matrix Spike D-Matrix Spike Duplicate X-Sample Duplicate

NOTE: Samples marked with an asterisk (*) required filtration after digestion and prior to analysis
Samples marked with a plus sign (+) required additional Conc.HNO3 in digestion process, brown fumes were observed

Reagents: 10mL 1:1 HNO3, Met 4475-07, Standards Log
5 mL Conc.HNO3, 6623E20033, Mallinckrodt
10mL 30% H2O2, 42H15, Mallinckrodt
10mL Conc.HCl, 5587C47A01, Mallinckrodt

Minimum digestion times have been met (analyst initials):

Hot Plate/Block Temp	Correction Factor

Digestate(s)	Date	Time	(Received)	Analyst	Location	Date	Time	(Relinquished)	Analyst	Location

	Lot Number	Manufacturer	
Initial Digestion Vessel:	A603LP021	Environmental Express	Pipet #: J0450H
Final Digestion Vessel	070603	Corning	Balance #: C94817
Filter Paper:	G1822665	Whatman	Printed on: 24-Aug-07 1:36:47 PM

Reviewed By: Date: MET-86-7232156

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APPENDIX C

CONTAMINATION CONTROL GUIDELINES

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APPENDIX C. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: ACID DIGESTION OF AQUEOUS SAMPLES

Method(s): SW846 Methods 3005A and 3010A and EPA Methods 200.7 and 200.8

Approvals (Signature/Date):

10/12/07	10/15/07
William Reinheimer Date	Steve Jackson Date
Technical Manager	Health & Safety Manager / Coordinator
10/12/07	10/12/07
Nasreen DeRubeis Date	Larry Matko Date
Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. PITT-IP-0003, Rev. 7.

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of aqueous samples for the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) and ICPMS using the MCAWW Method 200.7 (NPDES), EPA Method 200.8 and SW846 Methods 3005A and 3010A (RCRA).

1.2. The applicability of each of these preparation protocols to specific analytes is detailed in Tables I and II (Appendix A). Additional elements may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This SOP provides procedures applicable to the preparation of dissolved, total recoverable and total elements in surface water, ground water, aqueous samples, leachates/extracts.

1.4. SW-846 Method 3005A is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP or ICPMS.

1.5. ICP Method 200.7 and ICPMS Method 200.8 are used to prepare surface water, domestic and industrial waste samples for total recoverable and dissolved metals.

1.6. SW-846 Method 3010A is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis by ICP or ICPMS.

1.7. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

2. SUMMARY OF METHOD

2.1. Method 3005A/ Method 6010B ICP or 6020 ICPMS - Preparation for Total Recoverable or Dissolved Metals Analysis.

2.1.1. A representative aliquot of sample is heated with nitric and hydrochloric acids (concentrations and volumes differ between methods) and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to volume.

2.2. Method 3010A - Preparation for Total Metals Analysis by Method 6010B ICP or 6020 ICPMS.

2.2.1. A representative aliquot of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary) and brought up to volume.

2.3. Methods 200.7 and 200.8 have method specific preparations.

2.4. Digestion Procedures

2.4.1. The laboratory performs all the digestion procedures listed in the SOP, depending on the project requirements.

2.5. Refer to PITT-QA-0024 for subsampling procedures.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements that pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

3.5. Additional definitions of terms used in this SOP may be found in the glossary of the LQM.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix C for additional contamination control guidelines.

4.3. Boron and silica from the glassware will migrate into the sample solution during and following sample processing. For critical low-level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.7. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample.

4.8. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2. Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.3. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.4. 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.			

5.5. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit or under other means of mechanical ventilation.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor and the EHSC.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, hot block, or other adjustable heating source capable of maintaining a temperature of 90 - 95°C.

6.2. Thermometer that covers a temperature range of 0-150°C.

6.3. Hot Block Disposable Digestion Cups (from Environmental Express).

6.4. Watch glasses, plastic disposable.

6.5. Whatman No. 41 filter paper or equivalent.

6.6. Funnels or equivalent filtration apparatus.

6.7. Centrifugation equipment (if desired method of removing particulates is centrifugation).

6.8. Graduated cylinder or equivalent capable of measuring 50 mL within 3% accuracy.

6.9. Analytical balance capable of accurately weighing to the nearest 0.01 grams.

6.10. Repipetors or suitable reagent dispensers.

6.11. Calibrated automatic pipettes with corresponding pipet tips or Class A glass volumetric pipettes.

6.12. Class A volumetric flasks.

6.13. pH indicator strips (pH range 0 - 6).

6.14. Plastic digestate storage bottles.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom TestAmerica solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working ICP/ICPMS LCS/MS spike solution: The LCS/MS working spike solution is provided directly by the vendor, no further standard preparation is necessary.

7.4. The TCLP MS working spike solution is provided directly by the vendor, no further standard preparation is necessary.

7.5. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom TestAmerica solution, a solution must be purchased from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.6. Aqueous laboratory control samples (LCSW) and matrix spike samples are prepared as described in Sections 9.5 and 9.6. Refer to Tables III and IV (Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for ICP/ICPMS LCS and matrix spike preparations.

7.7. Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.8. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.10. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica is to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. For dissolved metals analysis, the samples should be filtered through a 0.45 um filter prior to preservation. Filtration must be done in the field or within 24 hours of collection.

Note: If a sample being analyzed for dissolved metals is found to contain sediment the analyst should contact their supervisor or group leader. The client should be notified of the problem to decide how to treat the sample.

9. QUALITY CONTROL

Table V (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using any method contained within this SOP the following requirements must be met:

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, which have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in PITT-QA-007. The spike level should be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the TestAmerica reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOPs (PITT-MT-0001 and PITT-MT-0020).

9.2. Preparation Batch - A group of up to 20 samples that is of the same matrix and is processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate (SW-846 Methods) or a matrix spike for every 10 or fewer sample (200.7). In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS, MSD) are not included in the sample count for determining the size of a preparation batch.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the

method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOP's. If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be redigested.

9.4.1. Aqueous method blanks are prepared by taking 50 mL or 50 g of reagent water through the appropriate procedure as described in Section 10.

9.4.2. TCLP method blanks are prepared by taking 50 mL or 50 g of leachate fluid through the appropriate procedure as described in Section 10.

9.5. Laboratory Control Sample (LCS) - One aqueous LCS (referred to as a Laboratory Fortified Blank in 200.7) must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOP's. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Refer to Section 7.3 for instructions on preparation of the aqueous LCS spike solution.

9.5.1. The aqueous LCS is prepared by spiking a 50 mL aliquot of reagent water with 0.5 mL of the working LCS/MS spike solution (7.3). The LCS is then processed through the appropriate procedure as described in Section 10.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch of up to 20 samples (SW-846 Methods) or one matrix spike is processed for every 10 or fewer samples (200.7). A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added (referred to as a Laboratory Fortified Matrix in 200.7). A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred.

9.6.1. The aqueous matrix spike sample is prepared by spiking a 50 mL aliquot of a sample with 0.5 mL of the working LCS/MS spike solution (7.3). The matrix spike sample is then processed as described in Section 10.

9.6.2. The TCLP matrix spike sample is prepared by spiking a 50 mL aliquot of a leachate with 0.5 mL of the working TCLP spike solution (7.4). The matrix spike sample is then processed as described in Section 10.

NOTE: The TCLP matrix spike must be added prior to preservation of the leachate.

9.6.3. If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria.

9.7. Quality Assurance Summaries - Certain clients may require specific project or program QC, which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. PROCEDURE

10.1. Hotplate/hotblock temperature must be verified daily for each hotplate/hotblock used and must be recorded on either the metals preparation log or in a hotplate/hotblock temperature logbook. The hotplate/hotblock temperature should be verified by measuring the temperature of a beaker or an equivalent digestion sample container of reagent water placed on each hotplate/hotblock.

10.2. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

10.3. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.4. All preparation procedures must be carried out in a properly functioning hood.

10.5. All samples are to be checked out of sample control with the chain of custody documentation filled out completely.

10.6. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample.

10.7. Samples are typically logged in as either waters or wastes. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment etc.) contact the lab supervisor or project manager for further instructions. In some cases it may be more appropriate to process these samples as solids.

10.8. If possible prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

10.9. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

10.10. The following procedure must be followed for all aqueous sample preparations:

10.10.1. Measure and record sample pH with pH paper on a separate aliquot of sample. This is typically verified and documented at sample receipt.

Note: If the sample pH is > 2 pH units, the client must be notified of the anomaly.

10.10.2. Mix sample by shaking the container.

10.10.3. Measure and transfer 50 mL or 50 g of the sample into a beaker.

Note: This SOP allows for samples to be weighed instead of measured volumetrically (See Section 18.1.2).

10.10.4. Measure extra aliquots of sample(s) selected for the MS or MS/MSD analysis. Spike each aliquot with 0.5 mL of spiking solution (7.3 or 7.4).

10.10.5. Measure and transfer 50 mL of reagent water into a beaker for the method blank.

10.10.6. Measure and transfer 50 mL of reagent water into a beaker for the LCS and add 0.5 mL of spiking solution (7.3)

10.11. Proceed to the appropriate Section for the desired method as follows:

Method 3005A	10.12
Method 3010A	10.13
Method 200.7	10.14
Method 200.8	10.15

10.12. **Method 3005A - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP/ICPMS (See Figure 1)**

10.12.1. To the sample beaker, add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl.

10.12.2. Cover with disposable watch glass.

10.12.3. Heat at 90 - 95°C until volume is reduced to between 15 and 20 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

10.12.4. Cool the beaker in a fume hood.

10.12.5. Wash down beaker walls and watch glass with reagent water.

10.12.6. Filter sample, if insoluble materials are present, though a prewashed (1% nitric acid) Whatman 41 filter paper or plunger filter into a disposable sample container.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

10.12.7. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

10.12.8. Adjust the final volume/mass to 50 mL or 50 g with reagent water. The sample is now ready for analysis

10.13. **Method 3010A - Preparation for Total Metals Analysis by ICP/ICPMS Spectroscopy (See Figure 2)**

10.13.1. To the sample beaker, add 1.5 mL of concentrated HNO_3.

10.13.2. Cover with disposable watch glass.

10.13.3. Place beaker on hotplate or hotblock (90-95 °C) and evaporate to low volume of 5 - 10 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

10.13.4. Cool the beaker in a fume hood.

10.13.5. Add another 1.5 mL portion of concentrated HNO_3 and re-cover the beaker.

10.13.6. Continue refluxing until the digestion is complete.

Note: Digestion is complete when the digestate is light in color or does not change in appearance. For most samples the addition of two nitric acid aliquots is sufficient, additional aliquots of nitric acid may be added if necessary.

10.13.7. Evaporate to low volume of 5 - 10 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry.

10.13.8. Cool the beaker in a fume hood.

10.13.9. Add 5 mL of 1:1 HCl.

10.13.10. Cover and reflux for an additional 15 minutes to dissolve precipitate or residue.

10.13.11. Wash down beaker walls and watch glass with reagent water.

10.13.12. Filter sample, if insoluble materials are present, though a prewashed (1% nitric acid) Whatman 41 filter paper or plunger filter into a disposable sample container.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

10.13.13. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

10.13.14. Adjust final volume/mass to 50 mL or 50 g with reagent water. The sample is now ready for analysis.

10.14. **Method 200.7 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICP (See Figure 3)**

10.14.1. To the sample beaker containing 50 mL of sample, add 1 mL of 1:1 HNO_3 and 0.5 mL of 1:1 HCl.

10.14.2. Heat at 80-85 °C until volume is reduced to between 15 and 20 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

10.14.3. Cover with disposable watch glass.

10.14.4. Gently reflux for 30 minutes.

10.14.5. Cool the beaker in the fume hood.

10.14.6. Wash down beaker walls and watch glass with reagent water.

10.14.7. Filter sample, if insoluble materials are present, though a prewashed (1% nitric acid) Whatman 41 filter paper or plunger filter into a disposable sample container.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

10.14.8. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

10.14.9. Adjust the final volume/mass to 50 mL or 50 g with reagent water. The sample is now ready for analysis.

10.15. **Method 200.8 - Preparation for Total Recoverable or Dissolved Metals Analysis by ICPMS (See Figure 4)**

10.15.1. To the sample beaker containing 100 mL of sample, add 2 mL of 1:1 HNO_3 and 1.0 mL of 1:1 HCl.

10.15.2. Heat at 80-85 °C until volume is reduced to between 15 and 20 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared

10.15.3. Cover with disposable watch glass.

10.15.4. Gently reflux for 30 minutes.

10.15.5. Cool the beaker in the fume hood.

10.15.6. Wash down beaker walls and watch glass with reagent water.

10.15.7. Filter sample, if insoluble materials are present, though a prewashed (1% nitric acid) Whatman 41 filter paper or plunger filter into a disposable sample container.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

10.15.8. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

10.15.9. Adjust the final volume/mass to 50 mL or 50 g with reagent water.

10.15.10. Take a 25 mL aliquot of the 50 mL sample volume and dilute up to 50 mL with de-ionized water.

10.16. Documentation and Record Management

10.16.1. The preparation benchsheet should, at a minimum, include the following information:

- Preparation date, analyst name, matrix, prep type), SOP reference.
- Sample ID, initial weight/volume and final weight/volume.
- Standards Documentation (source, lot, prep date, volume added).
- Analyst Signature.
- Reviewer's Signature and date.

11. CALCULATIONS / DATA REDUCTION

11.1. Not Applicable.

12. METHOD PERFORMANCE

12.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. In general, the matrix spike recovery should fall within +/- 25 % (SW-846 Methods) or +/- 30% (200.7) and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in determinative SOPs. The laboratory control samples should recover within 20% (SW-846 Methods) or 15% (200.7) of the true value until in house control limits are established. Acceptance criteria are given in the determinative SOPs. **Refer to PITT-QA-DoD-0001 for specific DoD QC requirements.**

12.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

12.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13. POLLUTION CONTROL

13.1. 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.

13.2. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

14. WASTE MANAGEMENT

14.1. The following waste streams are produced when this method is carried out.

14.1.1. Acidic waste containing nitric acid generated by the digestion. This waste is collected in a waste container identified as "Acid Waste", Waste #33. This waste is neutralized to a final pH between 6 and 9 and discharged down into a lab sink.

14.1.2. Contaminated disposable materials utilized for the analysis. These items are placed in trash containers which are emptied in the general trash dumpster located near the shipping/receiving dock.

15. REFERENCES

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A and 3010A.

15.2. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Method 200.7, Revision 4.4, May 1994.

15.3. Methods for the Determination of Metals in Environmental Samples, Supplement 1 (EPA/600/R-94/111), Method 200.8, Determination of Trace Elements in Waters by Inductively Coupled Plasma - Mass Spectrometry, Revision 5.4, 1994

15.4. PITT-MT-0001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010A and Method 200.7.

15.5. PITT-MT-0020, Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry (ICPMS) for Methods 200.8, 6020 & ILM05.2.

15.6. QA-003, TestAmerica QC Program.

15.7. QA-004, Rounding and Significant Figures.

15.8. PITT-QA-007, Method Detection Limits.

15.9. PITT-QA-0024, Subsampling.

15.10. PITT-QA-DoD-0001, Implementation of the DoD QSM Version 3.

16. ATTACHMENTS

16.1. Figure 1 – Method 3005A Flowchart

16.2. Figure 2 – Method 3010A Flowchart

16.3. Figure 3 – Method 200.7 Flowchart

16.4. Figure 4 – Method 200.8 Flowchart

16.5. Appendix A – Tables

16.5.1. Table I – Approved Preparation Method Analytes – SW846

16.5.2. Table II – Approved Preparation Method Analytes – NPDES

16.5.3. Table III – ICP/ICPMS Matrix Spike and Aqueous Laboratory Control Sample Levels

16.5.4. Table IV – TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

16.5.5. Table V – Summary of Quality Control Requirements

16.6. Appendix B – Metals Prep Benchsheet

16.7. Appendix C – Contamination Control Guidelines

17. REVISION HISTORY

17.1. Revision 7, 8/24/07

17.1.1. Changed laboratory name to TestAmerica.

17.1.2. Changed the format of the SOP to correspond to the new Corporate SOP format.

17.1.3. Added a reference for the source method for EPA Method 200.8.

17.1.4. For Method 200.7, removed the addition of 2.25 mL of HCl after the digestion.

17.1.5. For Method 200.7, revised the amount of 1:1 HCl added from 2.5 mL to 0.5 mL.

17.1.6. For Method 200.8, removed the addition of 1 mL of HCl after the digestion.

17.2. Revision 7.1, 10/11/07

17.2.1. For Method 200.7, removed the addition of 2 mL of concentrated HNO_3 after the digestion.

17.2.2. For Method 3005A, removed the addition of 1.5 mL of concentrated HNO_3 after the digestion.

18. METHOD MODIFICATIONS

18.1. Modifications applicable to SW-846 reference methods.

18.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client. **Refer to PITT-QA-DoD-0001 for specific DoD QC requirements.**

18.1.2. This SOP allows for aqueous samples to be weighed instead of measured volumetrically. This assumes the density of the sample is close to 1.0 g/mL. Samples with large amounts of sediment or suspended solids, sludges, non-aqueous liquids must be processed volumetrically. Weighing samples directly into the digestion vessel minimizes the potential for cross contamination, offers

improved accuracy over the use of graduated cylinders (comparable to volumetric flask accuracy), uses less glassware and is more efficient.

18.1.3. The referenced methods as well as Table 3-1 of SW-846 refer to the use of a 100 mL aliquot for digestion. This SOP requires the use of a 50 mL sample size to reduce waste generation. The use of reduced sample volumes are supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document stated "..flexibility to alter digestion volumes is addressed and "allowed" by the table (3-1) and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..." EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Olliver Fordham stated " As a "representative sample" can be assured, scaling causes no loss of precision and accuracy in the analysis."

18.2. Modifications Specific to Method 3010A

18.2.1. Section 10.13.7 of this SOP requires the sample be reduced to a volume of 5 - 10 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical analytes through volatilization.

18.2.2. The scope of 3010A has been expanded to include silver based on comparison studies with 7760A. Method 3010A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water and TCLP leachate) up to a concentration of 1 ppm silver.

FIGURE 1. METHOD 3005A

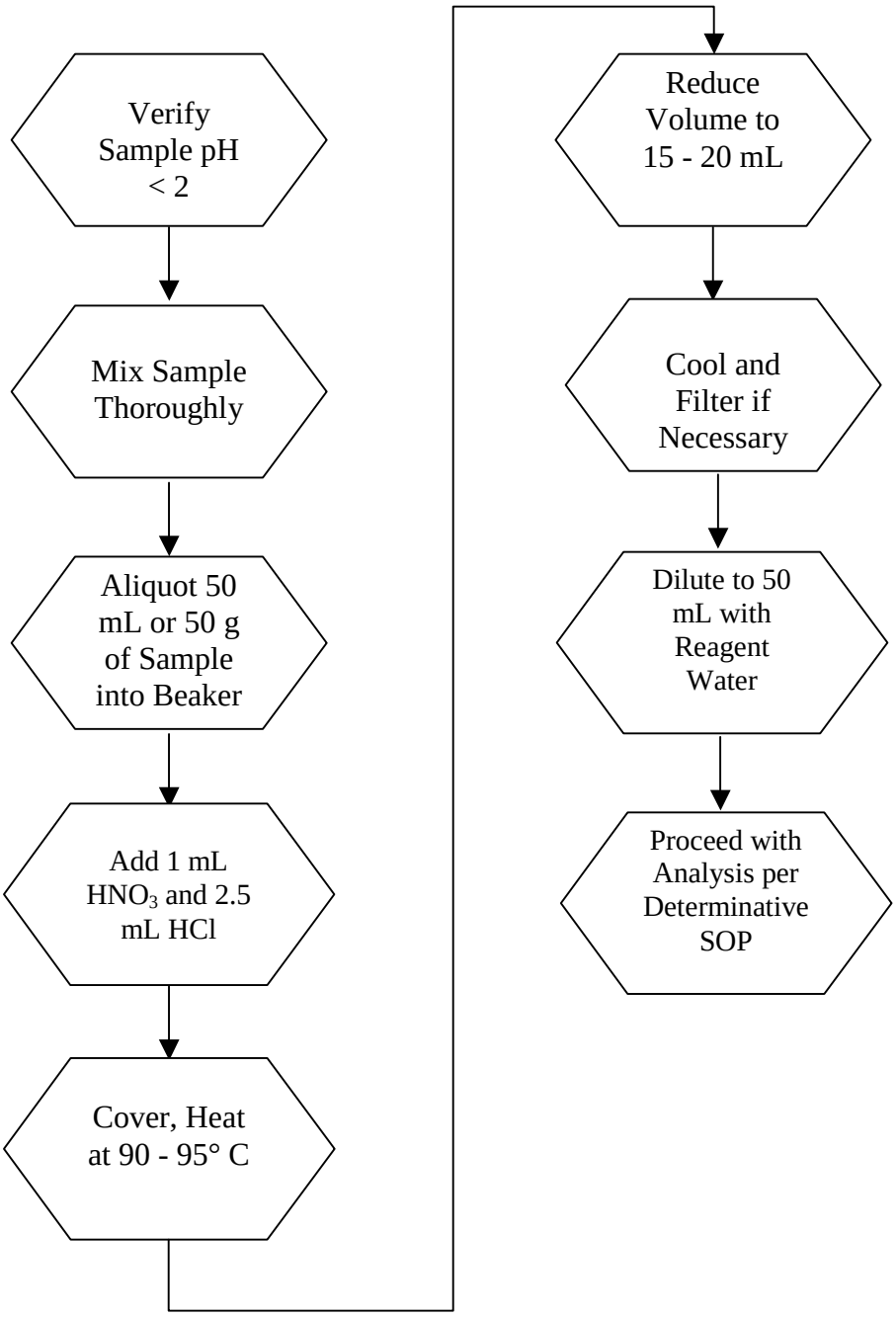


FIGURE 2. METHOD 3010A

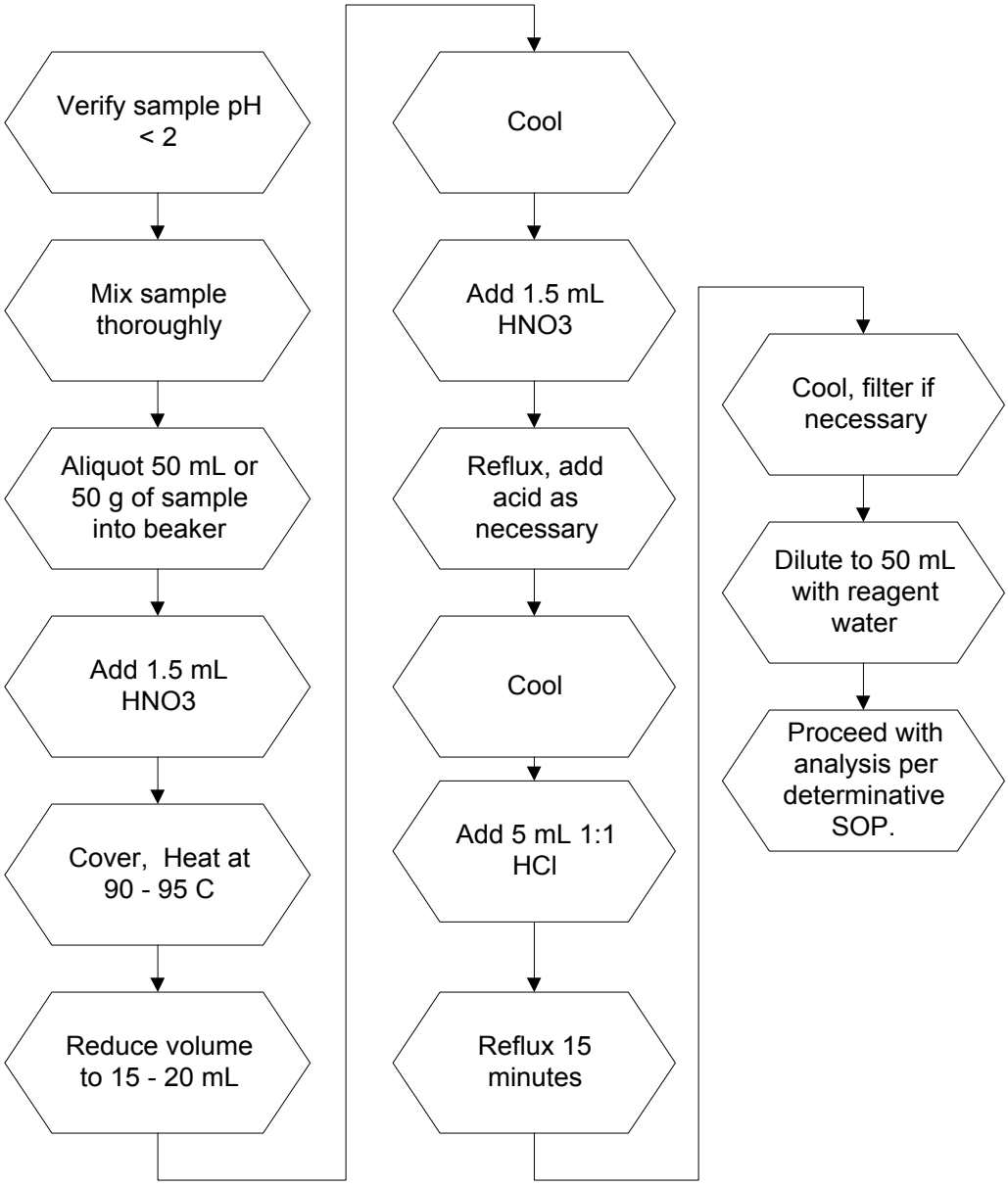


FIGURE 3. METHOD 200.7

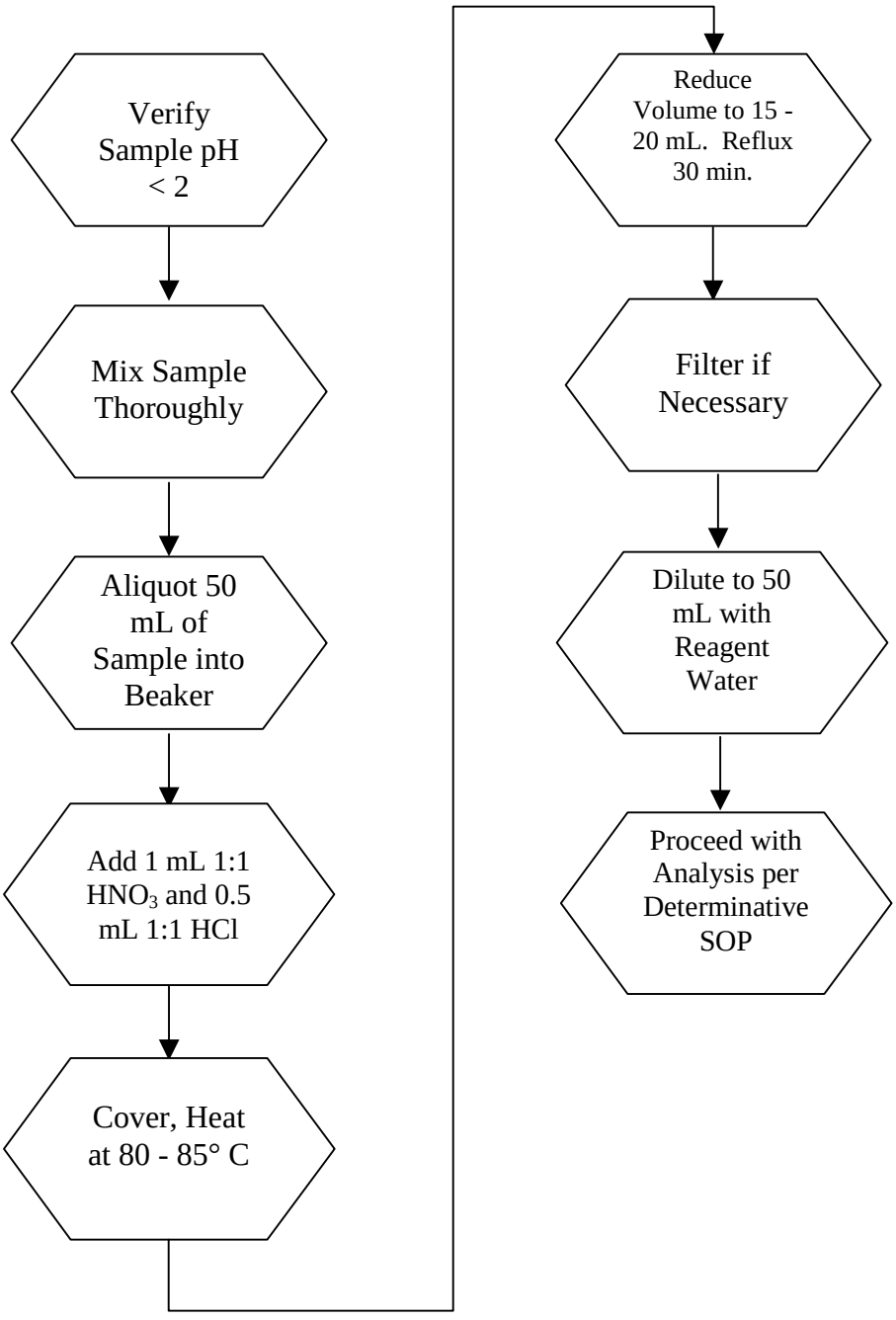
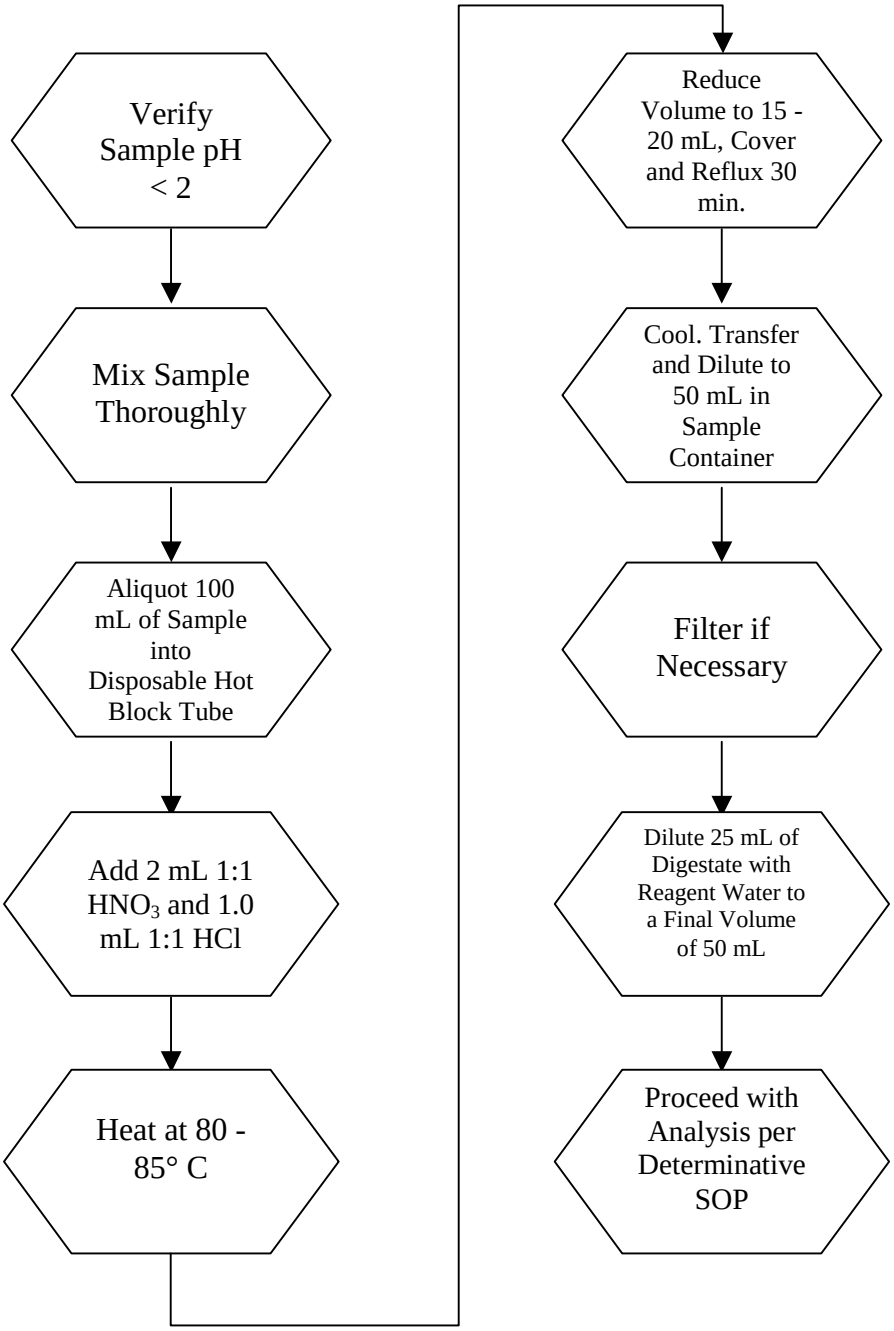


FIGURE 4. METHOD 200.8



APPENDIX A

TABLES

TABLE I
Approved Preparation Method Analytes - SW846

ELEMENT	Symbol	CAS Number	3005A	3010A
Aluminum	Al	7429-90-5	X	X
Antimony	Sb	7440-36-0	X	X
Arsenic	As	7440-38-2	X	X
Barium	Ba	7440-39-3	X	X
Beryllium	Be	7440-41-7	X	X
Cadmium	Cd	7440-43-9	X	X
Calcium	Ca	7440-70-2	X	X
Chromium	Cr	7440-47-3	X	X
Cobalt	Co	7440-48-4	X	X
Copper	Cu	7440-50-8	X	X
Iron	Fe	7439-89-6	X	X
Lead	Pb	7439-92-1	X	X
Magnesium	Mg	7439-95-4	X	X
Manganese	Mn	7439-96-5	X	X
Molybdenum	Mo	7439-98-7	X	X
Nickel	Ni	7440-02-0	X	X
Potassium	K	7440-09-7	X	X
Selenium	Se	7782-49-2	X	X
Silver	Ag	7440-22-4	X	X
Sodium	Na	7440-23-5	X	X
Thallium	Tl	7440-28-0	X	X
Vanadium	V	7440-62-2	X	X
Zinc	Zn	7440-66-6	X	X

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 12.0 of the SOP are met.

TABLE II
Approved Preparation Method Analytes - NPDES

ELEMENT	Symbol	CAS Number	200.7
Aluminum	Al	7429-90-5	X
Antimony	Sb	7440-36-0	X
Arsenic	As	7440-38-2	X
Boron	B	7440-42-8	X
Barium	Ba	7440-39-3	X
Beryllium	Be	7440-41-7	X
Cadmium	Cd	7440-43-9	X
Calcium	Ca	7440-70-2	X
Chromium	Cr	7440-47-3	X
Cobalt	Co	7440-48-4	X
Copper	Cu	7440-50-8	X
Iron	Fe	7439-89-6	X
Lead	Pb	7439-92-1	X
Magnesium	Mg	7439-95-4	X
Manganese	Mn	7439-96-5	X
Molybdenum	Mo	7439-98-7	X
Nickel	Ni	7440-02-0	X
Potassium	K	7440-09-7	X
Selenium	Se	7782-49-2	X
Silicon	Si	7631-86-9	X
Silver	Ag	7440-22-4	X
Sodium	Na	7440-23-5	X
Thallium	Tl	7440-28-0	X
Vanadium	V	7440-62-2	X
Zinc	Zn	7440-66-6	X

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 12.0 of the SOP are met.

TABLE III
ICP/ICPMS Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/ MS Level * (ug/l)
Aluminum	200	2000
Antimony	50	500
Arsenic	200 (ICP), 4 (ICPMS)	2000 (ICP), 40 (ICPMS)
Barium	200	2000
Beryllium	5	50
Cadmium	5	50
Calcium	5000	50000
Chromium	20	200
Cobalt	50	500
Copper	25	250
Iron	100	1000
Lead	50 (ICP), 2 (ICPMS)	500 (ICP), 20 (ICPMS)
Lithium	100	1000
Magnesium	5000	50000
Manganese	50	500
Molybdenum	100	1000
Nickel	50	500
Potassium	5000	50000
Selenium	200 (ICP), 1 (ICPMS)	2000 (ICP), 10 (ICPMS)
Silver	5	50
Sodium	5000	50000
Strontium	100	1000
Thallium	200 (ICP), 5 (ICPMS)	2000 (ICP), 50 (ICPMS)
Vanadium	50	500
Zinc	50	500
Boron	100	1000
Silica	1000	10000
Tin	200	2000
Titanium	100	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 0.5 mL working spike (7.3) to 50 mL of sample.

TABLE IV
TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE V
Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA[1]	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: PITT-MT-0001 and PITT-MT-0020	Redigest and reanalyze samples associated with the method blank.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: PITT-MT-0001 and PITT-MT-0020	Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples (SW-846 Methods) or one per every 10 or fewer samples (200.7).	Refer to determinative SOPs: PITT-MT-0001 and PITT-MT-0020	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: PITT-MT-0001 and PITT-MT-0020	See Corrective Action for Matrix Spike.

[1] **For specific DoD requirements, refer to PITT-QA-DoD-0001.**

APPENDIX B

METALS PREP BENCHSHEET

METALS PREP BENCHSHEET

TestAmerica Pittsburgh ***Metals Preparation Log*** Batch No. 7232156 Page 1 of 1

v1.0.6 - Last Revised 12/30/2005

Method 3050B	Matrix Solid	Start Time 0945	SDG -
Analyst example	Date 082007	Lot Numbers	
MS STLPA-MS-A, STLPA-MS-B, STLPA-MS-C		C7H030157	
Lab Lot # C7H030157			
Analyst Sign:			

Sample ID	Initial Wt/Vol g/mL	Final Vol mL	Comments
J3500			
J3501			
J3503			
J3505			
J3507			
J3509			
J350A			
J350C			
J350F			
J350H			
J350K			
J350N			
J350R			
J350W			
J351D			
J35X4			
J35X6			
J35X8			
J35X9			
J35XM			
J35XM S			
J35XM D			
J47LA B			
J47LA C			

SAMPLE CODING: B-Blank C-Check L-Check Duplicate S-Matrix Spike D-Matrix Spike Duplicate X-Sample Duplicate

NOTE: Samples marked with an asterisk (*) required filtration after digestion and prior to analysis
Samples marked with a plus sign (+) required additional Conc.HNO3 in digestion process, brown fumes were observed

Reagents: 10mL 1:1 HNO3, Met 4475-07, Standards Log
5 mL Conc.HNO3, 6623E20033, Mallinckrodt
10mL 30% H2O2, 42H15, Mallinckrodt
10mL Conc.HCl, 5587C47A01, Mallinckrodt

Minimum digestion times have been met (analyst initials):

Hot Plate/Block Temp	Correction Factor

Digestate(s)	Date	Time	(Received)	Analyst	Location	Date	Time	(Relinquished)	Analyst	Location

	Lot Number	Manufacturer	
Initial Digestion Vessel:	A603LP021	Environmental Express	Pipet #: J0450H
Final Digestion Vessel	070603	Corning	Balance #: C94817
Filter Paper:	G1822665	Whatman	Printed on: 24-Aug-07 1:36:47 PM

Reviewed By: Date: MET-86-7232156

APPENDIX C

CONTAMINATION CONTROL GUIDELINES

Controlled Source: Intranet

This is a Controlled Document. When Printed it becomes Uncontrolled.

APPENDIX C
CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with nitric acid prior to routine cleaning.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Semivolatile Organic Analysis by GCMS

Method(s): 8270C and 625

Approvals (Signature/Date):

Sharon Bacha Technical Manager	11/16/07 Date	Steve Jackson Health & Safety Coordinator	11/16/07 Date
Nasreen K. DeRubeis Quality Assurance Manager	11/16/07 Date	Larry Matko Laboratory Director	11/16/07 Date

This SOP was previously identified as SOP No. PITT-MS-0001, Rev. 7

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1. SCOPE AND APPLICATION

1.1. This method is based upon SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. The modifications presented in Attachment A may be followed for analysis of wastewater following method 625. Direct injection of a sample may be used in limited applications. Refer to Tables 1 through 4 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.

1.2. The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Hexachlorophene is not amenable to analysis by this method.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

1.3. The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1

and 2 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

1.4. For DoD QSM Version 3 additional requirements, refer to SOP PT-QA-025.

1.5. Analytes, Matrix(s), and Reporting Limits:

1.5.1. This method is used to determine semivolatile organic compounds in a variety of matrices: water, soil, sediment, sludge, waste and tissue samples.

1.5.2. Reporting Limits are listed in Tables 1 through 2D.

2. SUMMARY OF METHOD

2.1. Aqueous samples are extracted with methylene chloride using a separatory funnel, a continuous extractor or Accelerated One-Step™. Solid samples are extracted with methylene chloride / acetone using sonication, soxhlet, accelerated soxhlet or pressurized fluid extraction. Waste dilution is used for samples that are miscible with the solvent. The extract is dried and concentrated to a final volume as defined for the matrix in the extraction SOP. Extraction procedures are detailed in SOP# PT-OP-001. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3. DEFINITIONS

3.1. CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.

3.2. SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3. Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Batches are defined at the sample preparation stage.

Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica Pittsburgh QC Program document (QA-003/PT-QA-021) for further details of the batch definition.

3.4. Method Blank - An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.5. LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.6. MS (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.

3.7. MSD (Matrix Spike Duplicate)- a second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method.

3.8. PT-LQAM – Pittsburgh laboratory quality assurance manual.

3.9. Method Code QL – Quantims (LIMS) Method code for 8270C.

3.10. Method Code 42 – Quantims (LIMS) Method code for 8270C low level.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2. The use of high purity reagents, solvents, and gases helps to minimize interference

problems.

4.3. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4. Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5. Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2. 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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.4. Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica Pittsburgh associate. The situation must be reported immediately to a laboratory supervisor or EH&S coordinator

6. EQUIPMENT AND SUPPLIES

6.1. Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/split less injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2. Column: 30 m x 0.32 mm I.D. (or 0.25 mm I.D.) 0.5-µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3. Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when 50 ng of the GC/MS tuning standard is injected through the GC.

6.4. GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.5. Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of

plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.6. Syringe: 10 µL Hamilton Laboratory grade syringes or equivalent.

6.7. Carrier gas: Ultra high purity helium.

7. REAGENTS AND STANDARDS

7.1. A minimum of seven calibration points are prepared. The low point should be at or below the reporting limit. Refer to Tables 12 through 13 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2. An Internal Standard solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10. The standard is stored at -10ºC ± 2 ºC.

7.2.1. Internal Standards are added to all standards and extracts to result in 40ng injected onto the column. For example, if the volume of an extract used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added for a 1 µL injection. For low level analysis internal standards are added to all standards and extracts to result in 8 ng injected onto the column. For example, if the volume of an extract being analyzed is 100 uL, 1 µL of a 400 µg/mL internal standard solution would be added for a 2 µL injection.

7.3. Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.

7.4. GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at 50 µg/mL. The standard is stored according to manufacturer recommendations.

7.5. Laboratory Control Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. LCS compounds and levels are listed in Tables 9 and

10.

7.6. Matrix Spike Solution: Prepare as indicated in the preparative methods. See preparation SOP. The matrix spike compounds and levels are the same as the LCS compounds.

7.7. The standards listed in 7.1 to 7.6 should be refrigerated at $\leq 6^{o}C$ when not in use. Refrigeration at -10°C to -20°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year. The continuing calibration standard must be replaced every week and is stored at 4 °C ± 2°C.

7.8. Standard Stock Solutions: See attachment "Standard Preparation Logbook Record".

8. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1. Reference appropriate facility SOPs and LQAM for sample bottle preservation.

8.2. Samples are stored at 4 ± 2°C. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. The extracts are stored at -10 ºC ± 2ºC (Extracts will normally be stored for 30 days after invoicing.)

8.3. Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

9. QUALITY CONTROL

9.1. See Document QA-0003 "TestAmerica Pittsburgh Quality Control Program" for additional detail. **For DoD QSM requirements and exceptions to requirements refer to SOP PT-QA-025 and Table B-1 and B-3**.

9.2. Initial Demonstration of Capability

9.2.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin. Refer to the flow chart in section 17.2.

9.2.2. For non-standard analytes an MDL study should be performed and calibration curve generated before analyzing any samples, unless lesser requirements are

previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.3. Control Limits

For DoD quality control requirements and acceptance criteria see SOP PT-QA-025. In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, MS and LCS Precision limits for matrix spikes / matrix spike duplicates are mean relative percent difference +/- 3 standard deviations.

9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 10X.

9.3.2. Routine 8270, QL and 42 Method Codes - Surrogates will be considered DIL, NC (Diluted out – can not be calculated) at 11X or above. Any dilution between a straight run and 10X run will be reported. Straight runs up to a 5X we should be able to see surrogates. If surrogates are outside QC limits and no obvious matrix is visible, these samples will go back for reextraction provided there are no technical reasons why reextraction should not be done. Project Manager approval will be required if technical judgment is used not to reextract. If surrogates are outside QC for dilutions 6X through 10X a NCM will be generated noting surrogates are out due to dilution.

9.3.3. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3.4. Refer to the QC program document (QA-003/PT-QA-021) for further details of control limits.

9.4. Method Blank

9.4.1. A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples, and sodium sulfate

for soil samples (Refer to SOP No. PT-OP-001 for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher. Refer to the TestAmerica Pittsburgh QC Program document (QA-003/PT-QA-021) for further details on the corrective actions. **For DoD requirements see PT-QA-025, Implementation of the DoD QSM Versions 3, January 2006**.

- If the analyte is a common laboratory contaminant (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.

- Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.4.2. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.4.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.4.4. Sample results are NOT to be blank subtracted.

9.5. Instrument Blank

9.5.1. Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.6. Laboratory Control Sample (LCS)

9.6.1. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. The LCS is spiked with all target compounds listed unless specified otherwise by a client or agency. All control analytes must be within established control limits (Table 9). The compounds must be spiked at a concentration appropriate for the chosen method of analysis, see Tables 9 through 10 for routine 8270 and low level (method codes 42 and QL). For DoD LCS control limits and requirements see SOP PT-QA-025.

9.6.2. If any control analyte (Table 9) in the LCS is outside the laboratory established historical control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. The analyst should consult with the PM and QA Manager to ensure that reporting with narration is acceptable with the client and program. Where this is approved a non-conformance memo will be created including all evidence that the associated samples are not affected.

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6.3. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every

batch of samples. The MS/MSD is spiked with the same analytes as the LCS (full analyte spike). Compare the percent recovery and relative percent difference (RPD) of the control analytes to that in the laboratory specific historically generated limits. (Table 9)

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.8. Surrogates

9.8.1. Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. Surrogate compounds must be spiked at appropriate level chosen for the method of analysis, see Table 11 for 8270 routine and low level surrogates. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.8.2. If any surrogates are outside control (Table 15) limits the following corrective actions must take place (except for dilutions):

- Check all calculations for error.

- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.
- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.8.3. If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

9.8.4. If the surrogates were within control limits in the sample and the MS/MSD surrogates are outside QC limits, the MS/MSD confirm matrix interference and sample and MS/MSD will not be reextracted. If the surrogates are outside QC limits in the sample but the MS/MSD surrogates are within QC limits, the sample will be reextracted and reanalyzed. If there is a trending pattern with the samples, analyst will use technical judgment whether to reextract or not.

9.8.5. If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.8.6. If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.8.7. Routine 8270, QL and 42 Method Codes - Surrogates will be considered DIL, NC (Diluted out – can not be calculated) at 11X or above. Any dilution between a straight run and 10X run will be reported. Straight runs up to a 5X we should be able to see surrogates. If surrogates are outside QC limits and no obvious matrix is visible, these samples will go back for reextraction provided there are

no technical reasons why reextraction should not be done. Project Manager approval will be required if technical judgment is used not to reextract. If surrogates are outside QC for dilutions 6X through 10X a NCM will be generated noting surrogates are out due to dilution.

9.8.8. **For DoD work the QSM indicates that all surrogate exceedances must be re-prepped and reanalyzed for confirmation of all matrix effects**.

9.9. Nonconformance and Corrective Action

9.9.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.10. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.11. TestAmerica Pittsburgh QC Program

Further details of QC and corrective action guidelines are presented in the TestAmerica Pittsburgh QC Program documented in Policy QA-003/PT-QA-021.

10. PROCEDURE

CALIBRATION AND STANDARDIZATION

10.1. **For DoD QSM Version 3 calibration requirements refer to SOP PT-QA-025**.

10.2. Summary

10.2.1. The instrument is tuned for DFTPP, calibrated initially with a six-point calibration curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 5.

10.3. All standards and extracts are allowed to warm to room temperature before injecting.

10.4. Instrument Tuning

At the beginning of every twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) is achieved for DFTPP (decafluorotriphenylphosphine).

10.4.1. Inject 50 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. Acceptable means of passing DFTPP are as follows:

- The peak apex, or the scan immediately before the apex, or the scan immediately after the apex, or the average of these three scans may be used. The average of the apex and the scan before or after the apex may also be used.
- Background subtraction is optional. If background subtraction is used, a single scan must be subtracted and this scan cannot contain any significant ions of 198 of 442. This single scan to be subtracted must be prior to and within 20 scans of the start of DFTPP elution and it must not be part of the DFTPP peak.
- If the instrument has a built in macro that checks the DFTPP, use of this macro with no manual manipulation is also acceptable and preferred (assuming , of course that the correct ion ratios are being checked).
- If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.4.2. The GC/MS tuning standard must also be used to evaluate the inertness of the chromatographic system. The tailing factor for Benzidine and pentachlorophenol must be calculated. Benzidine must have a tailing factor that is less than 3 and pentachlorophenol must have a tailing factor that is less than 5. If DDT is an analyte of interest, it must be included in the tuning standard, and its breakdown must be ≤ 20%. The DDT breakdown check minimum frequency is daily prior to analysis of samples. The entire calculation must be included on the raw data. Refer to section 12 for the appropriate calculations.

10.5. Initial Calibration

10.5.1. Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If

interferences are noted, use one of the next two most intense masses for quantitation. For DoD initial calibration requirements refer to SOP PT-QA-025.

10.5.2. Compounds should be assigned to the IS with the closest retention time.

10.5.3. Prepare calibration standards at a minimum of seven concentration levels for each target compound and all surrogates. Six standards must be used for a quadratic least squares calibration. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response. In either case, the lowest standard must be at or below the reporting limit and the use of only five standards requires a linear curve technique to be used. Add the internal standard mixture to result in 40 ng on column. (For example, if the volume of the calibration standard used is 1 mL, add 100 µL of the 400 µg/mL internal standard solution for a 1 µL injection). The concentrations of all analytes are listed in tables 12 and 13. For low level analysis internal standards are added to all standards and extracts to result in 8 ng injected onto the column. For example, if the volume of an extract being analyzed is 100 uL, 1 µL of a 400 µg/mL internal standard solution would be added for a 2 µL injection.

10.5.4. Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in section 12 and verify that the CCC and SPCC criteria in section 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**

10.5.5. System Performance Check Compounds (SPCCs): The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene

2,4-Dinitrophenol
4-Nitrophenol

10.5.6. Calibration Check Compounds (CCCs): The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

10.5.6.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.6.2. CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.5.7. Note: the laboratory may not use the "grand mean" rule. The following are guidelines that are used for routine SW-846 analysis within the laboratory, however these guidelines are subject to program and project specific requirements.

10.5.8. Where a target compound is ≤15% RSD an average response factor curve may be used. If the 15% RSD criteria is exceeded for a non-CCC target compound the analyst must assess the curve and attempt to apply a "best-fit" curve function. The first step of the assessment is to find out if the quadratic curve will have a correlation coefficient of ≥ .995. If it does not, then use the average response factor. If it does, then review where the quadratic curve intercepts the

y- axis in comparison to the MDL and origin. Also review the shape of the curve. Does it overlap itself or have other potential problems? These steps should all be used in deciding when a quadratic curve or average response factor curve would be best.

10.5.9. Where a quadratic or polynomial curve is used R must be ≥.995 for a curve to be considered to be an acceptable fit.

10.5.10. All linear curves for non-CCC compounds that exceed 15% RSD or best-fit curve functions that have R < .995 are in exceedance of guidance criteria and must be evaluated for corrective action. Any non-CCC compound being reported from a curve that does not meet either the 15% RSD criteria or the R = .995 for a "best-fit" curve will be narrated as a non-conformance.

10.5.11. The following exceptions may be reportable with narration depending on the project DQO's and data usability requirements:

10.5.11.1. Where a target compound is ≥15% but ≤30% an average response factor curve may still be used if the analyst shows that the average response factor is an acceptable fit over the range of use. A graphical representation of the curve should be presented for documentation. However, if the quadratic curve is clearly a better fit it should be used.

10.5.11.2. Compound list will be divided into two lists: List 1 (reliable performers) and List 2 (poor performers). List 1 compounds should always have a %RSD less than 30% or correlation coefficient of .995 with an allowance of up to four sporadic marginal failures for semivolatiles. Sporadic marginal failures for these compounds should be </= 40% or .990. Sporadic marginal failures require a print out of the curve with narration.

10.5.11.3. List 2 compounds is comprised of the list of known poor performers. For List 2 analytes, where the %RSD is ≤15% an average response factor will be used. For %RSDs >15% and ≤60% the best fit curve will be selected. For these compounds a print out of the curve will be provided as a graphical documentation of curve performance.

10.5.11.4. Documentation: Raw target curve summary with all compounds set to average response factor will be provided. If quadratic or polynomial equations are used a reprint of the curve table will be provided to show the correlation coefficient for the "best fit" equation. And as noted above, compounds that need additional documentation to demonstrate the curve fit will have a graphical presentation of the curve provided for reference.

10.5.11.5. Any analyte not on List 1 or List 2 would be held to specific criteria based on project specific requirements.

10.5.11.6. Any non-CCC compound being reported from a curve that does not meet either the 15% RSD criteria or the R = .995 for a "best-fit" curve will be narrated as a non-conformance.

10.5.11.7. All %RSDs that are >30% must be narrated and when using an average response factor curve for a %RSD >30 % should also be narrated.

10.5.12. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/Concentration^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.5.13. If time remains in the 12 hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.5.14. **Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.**

10.5.15. **Second Source Calibration Verification Requirements:**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 30% of expected value (initial source) for all work except DoD. For DoD work all analytes must be within ± 25% of expected value. The exception to this requirement is for poor performers: bis- 2- chloroisopropyl ether, Benzoic Acid, 2,4 Dinitrophenol, 2-Naphthylamine, Benzaldehyde and Pentachlorophenol. For these compounds the criteria is 50-150. **Note:** 2-Naphthylamine and Benzaldehyde are not DoD compounds.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

10.6. Continuing Calibration

10.6.1. At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 6.

10.6.2. Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.

10.6.3. The following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of ≥ 0.05.

- The percent difference or drift of the CCC compounds from the initial calibration must be ≤ 20%. (see section 12 for calculations)
- List 1 compounds that are Non CCC's must be ≤ 25% differences or drift with the allowance of up to four which must be ≤ 40% .
- List 2 target compounds including Appendix IX will be accepted where the % difference or drift is ≤ 50%.
- Where a List 2 target compound is out high by > 50% and the compound is ND in the samples, the samples may be reported with narration.
- If a list 1 compound is not found in the sample, a CCV(out high) of up to 50%D or drift, may be accepted with narration subject to determination that it is acceptable for the specific project.
- Any compound with a %D or Drift >25% must be narrated.
- The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.
- The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.

10.6.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.6.4. Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

10.7. **Sample Preparation**

Samples are prepared following SOP PT-OP-001.

10.8. **Sample Analysis Procedure**

10.8.1. Calibrate the instrument as described in section 10. Depending on the target

compounds required by the client, it may be necessary to use more than one calibration standard.

10.8.2. All samples must be analyzed using the same instrument conditions as the preceding continuing calibration standard.

10.8.3. Add internal standard to the extract to result in 40 ng injected on column (for example, 1 µL of a 2000µL/mL internal standard solution in 100 µL of extract for a 2 µL injection). Mix thoroughly before injection into the instrument. For low level analysis internal standards are added to all standards and extracts to result in 8 ng injected onto the column. For example, if the volume of an extract being analyzed is 100 uL, 1 µL of a 400 µg/mL internal standard solution would be added for a 2 µL injection.

10.8.4. Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

10.8.5. The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.

10.8.6. Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system. For manual integration practices refer to TestAmerica corporate SOP, S-Q-004, Acceptable Manual Integration Practices. For DoD and all other projects the following criteria must be met:

When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of manual integration (i.e., chromatograms of manually integrated peaks), and notation of rationale, date, and name or initials of person performing manual integration operation (electronic signature is acceptable). DoD QSM, Version 3, Clarification 50 and 57.

Case Narrative. For DoD the case narrative shall provide: identification of **samples and analytes** for which manual integration was necessary. DoD QSM, Version 3, Appendix DoD-A.

10.8.7. Target compounds identified by the data system are evaluated using the criteria

listed in section 11.0.

10.8.8. Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in section 11.0. At least 20 TICs will be generated.

10.9. Tissue analysis follows the same procedure as other samples as described in this SOP.

10.10. Initial review and corrective actions

10.10.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.10.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatography will be reviewed and if in the technical judgment of the analyst obvious matrix interference is observed and the chromatographic system returns within control, samples will be reported as is if not reanalysis of samples analyzed while the system was malfunctioning is required.

10.10.3. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. Samples that are reported with internal standard exceedances must have documentation supporting matrix effect. Where the matrix effect is well established it may be reported with narration, otherwise the samples must be reanalyzed to confirm matrix effect is required. If the internal standard exceedance is deemed to be due to an instrumental problem, instrument maintenance will be done and all affected samples must be reanalyzed after the problem is corrected

10.10.4. The surrogate standard recoveries are evaluated to ensure that they are within limits. See section 9.8 for corrective actions for surrogate recoveries.

10.11. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a

dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, based on analyst technical judgment, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.11.1. Routine 8270, QL and 42 Method Codes - Surrogates will be considered DIL, NC (Diluted out – can not be calculated) at 11X or above. Any dilution between a straight run and 10X run will be reported. Straight runs up to a 5X we should be able to see surrogates. If surrogates are outside QC limits and no obvious matrix is visible, these samples will go back for reextraction provided there are no technical reasons why reextraction should not be done. Project Manager approval will be required if technical judgment is used not to reextract. If surrogates are outside QC for dilutions 6X through 10X a NCM will be generated noting surrogates are out due to dilution.

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. If viscosity of the sample is in question, as per analyst technical judgment, the lowest possible dilution will be done in order for the autosampler to function properly due to viscosity. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

10.11.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

10.12. Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 $\pm$ 2°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

10.13. Retention time criteria for samples

Retention time windows must be established and verified once per ICAL and at the beginning of the analytical shift as per DoD QSM, Version 3, Appendix DoD-B, Table

B-3. If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.13.1. If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
Retention Time window position establishment for each analyte and surrogate	Once per ICAL	Position shall l be set using the midpoint standard of the initial calibration curve.	NA	NA
Evaluation of relative retention times (RRT)	With each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.

10.14. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to the facility specific SOP for determination of percent moisture.

10.15. Procedural Variations

10.15.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. Any unauthorized deviations from this procedure must also be documented as a non-conformance, with a cause and corrective action described.

10.16. Troubleshooting Guide

10.16.1. Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the TestAmerica Pittsburgh Laboratory Quality Assurance Manual (LQAM), the following daily maintenance should be performed.

10.16.1.1. Clip Column as necessary.

10.16.1.2. Install new or cleaned injection port liner as necessary.

10.16.1.3. Install new septum as necessary.

10.16.1.4. Perform mass calibration as necessary.

10.16.2. Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

11. CALCULATIONS / DATA REDUCTION

11.1. Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards, referencing the hardcopy “clean” spectra reference book or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component relative retention time must compare to ± 0.06 RRT units of the retention time of the standard component. For reference, the

standard must be run within the same twelve hours as the sample.

- All ions present in the standard mass spectra at a relative intensity greater than 30% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

- The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

- The relative intensities of ions should agree to within ±30% between the 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%.)

11.1.1. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

11.2. Mass chromatogram searches:

Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte a mass chromatogram search is made.

11.2.1. Hexachlorophene

Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

11.3. 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 type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification.

Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- 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 should be between 30%and 70%.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.
- 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 coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

11.4. Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

The compounds which may be analyzed by 8270C include some problem compounds

would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

11.5. Calculations

11.5.1. Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

$\overline{RF}$ = Mean of RFs from intial caibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$\text{SD} = \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

RFi = RF for each of the calibration levels

N = Number of RF values

11.5.2. Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

11.5.3. Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

11.5.4. Average response factor

If the average of all the %RSDs of the response factors in the initial calibration is $\leq$ 15%, the average response factor from the initial calibration may be used for quantitation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x} \qquad mean\ \overline{RF} = \sum_{i=1}^{n} RF_i / n$$

Where:

$\mathbf{A_x}$ = Area of the characteristic ion for the compound to be measured

$\mathbf{A_{is}}$ = Area of the characteristic ion for the specific internal standard

$\mathbf{C_{is}}$ = Concentration of the specific internal standard, ng

$\mathbf{C_x}$ = Concentration of the compound being measured, ng

11.5.5. **Relative Retention Time (RRT)** – is the ration of the retention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RT_C}{RT_{is}}$$

Where,

RT_C = Retention time for the volatile tragert compounds in the continuing calibration.

RT_{is} = Retention time for the internal standard in calibrtion standard or in a sample.

11.5.6. Linear fit

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

C_{ex}= Concentration in extract, µg/mL

R_x= Response for analyte

R_{is}= Response for internal standard

C_{is}= Concentration of internal standard

A= Intercept

B= Slope

11.5.7. Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

C = Curvature

11.5.8. The concentration in the sample is then calculated:

11.5.8.1. Aqueous Calculation

$$\text{Concentration, } \mu\text{g / L} = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (mL)

C_{ex} = *Result from linear or quadratic fit*

11.5.8.2. Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

11.5.9. MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

S_{SR} = Spike sample result

S_R=Sample result

S_A = Spike added

11.5.10. Relative % Difference calculation for the MS/MSD

$$RPD = \frac{\left| MS_R - MSD_R \right|}{(MS_R + MSD_R)/2} \times 100$$

RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

11.5.11. Relative response factor calculation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

A_x=Area of the characteristic ion for the compound being measured

A_{is}=Area of the characteristic ion for the specific internal standard

C_x=Concentration of the compound being measured (µg/L)

C_{is} =Concentration of the specific internal standard (µg/L)

11.5.12. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x=Area of the total ion chromatogram for the compound being measured

A_{is}=Area of the total ion chromatogram for the nearest internal standard without interference

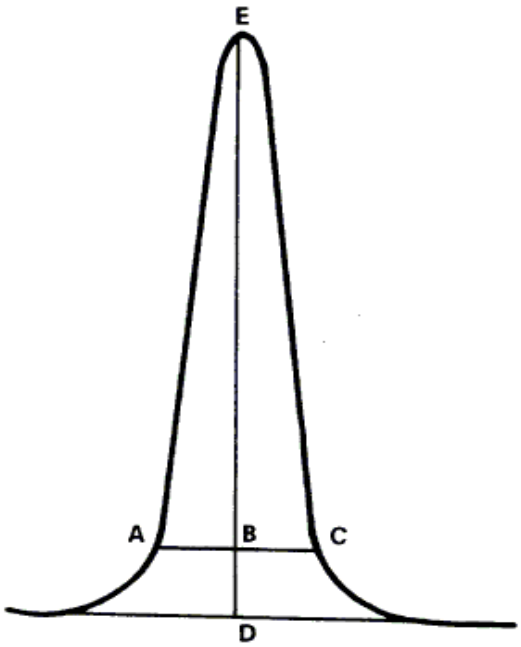
RF=1

11.5.13. Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

11.5.14. Tailing Factor Calculation



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

12. METHOD PERFORMANCE

12.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP # PITT-QA-0007. MDLs for the analytes of interest are performed as per SOP PITT-QA-0007.

12.2. Initial Demonstration

Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. IDOC is analyzed for each new analyst.

12.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the level 3 calibration standard.

12.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in tables 14 and 14A.

12.2.3. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4. The CCV will be varied periodically to demonstrate verification of linearity of the curve.

12.3. Non-standard analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

12.4. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

12.5. Data Quality Objectives (DQO). Refer to project-specific Quality Assurance plans for DQO information.

13. POLLUTION CONTROL

13.1. 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. WASTE MANAGEMENT

14.1. The following waste streams are produced when this method is carried out.

14.1.1. Solvent waste generated from cleaning operations and out of specification standards. This waste is placed in a waste container identified as "Methylene Chloride Waste", Waste #2 or "Mixed Flammable Solvent Waste", Waste #3.

14.1.2. Sample extracts in vials. This waste is placed in containers identified as "Vials & Extracts", Waste #7.

14.1.3. Sylon Waste. This waste is collected in a container identified as "Sylon (5%) / TolueneWaste", Waste #20.

15. REFERENCES / CROSS-REFERENCES

15.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update II, October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

15.2. J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975).

15.3. SOP # PT-QA-025, Implementation of DoD QSM Version 3 January 2006, current version.

15.4. USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, OSWER 9240.1-05A-P, PG99-963-506, EPA540/R-99/008, October 1999.

15.5. SOP # PT-OP-001, Extraction and Cleanup of Organic Compounds from Waters and

Solids, based on SW-846 3500 series, 3600 series, and Method 8151A.

15.6. SOP # PITT-QA-0007, Determination of Method Detection Limits (MDL).

15.7. SOP # S-Q-004, Acceptable Manual Integration Practices.

15.8. Pittsburgh Laboratory Quality Assurance Manual (PT-LQAM).

16. METHOD MODIFICATIONS

16.1. Modifications from Reference Method

16.1.1. A relative retention time window of ± 0.06 RRT units is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

16.1.2. The quantitation and qualifier ions for some compounds have been added to the list of those which are recommended in SW-846 in order to improve the reliability of qualitative identification.

17. ATTACHMENTS

17.1. Attachment A - Modifications Required For Analysis Of Wastewater Following Method 625

17.2. Appendix A - Routine Calibration Criteria For Most Projects Using SW-846 8270C – For DoD refer to DoD SOP PT-QA-025.

17.3. Attachment B – Standard Preparation Logs

17.4. Appendix B – EPA Memo Regarding Method 625 Modifications

17.5. Appendix C – DoD QSM QA/QC Requirements

18. REVISION HISTORY

18.1. Modifications in this version of SOP are highlighted throughout the procedure.

18.2. 8270C low level analysis added to this SOP. Calibration levels, internal standard levels and spike levels, dilution requirements and reporting limits were all updated for method

codes 42 and QL.

18.3. SOP format updated to TestAmerica SOP format.

Appendix A – Calibration Criteria

This Appendix summarizes routine calibration criteria for most projects using SW-846 8270C. It is superceded by project specific requirements that may specify project specific DQOs. The purpose of this section is to identify exceedances, which are typically reportable with narration for most projects, and exceedances, which are not normally reportable except with permission of the client in advance. The criteria presented are based on SW-846 and national functional guidelines for data validation and data usability. This document is also written into a work instruction. **For DoD requirement refer to SOP PT-QA-025**.

INITIAL CALIBRATION

Number of Points

1) A five-point curve is required for use of average response factor.

2) A six-point curve is required for use of quadratic curves.

3) A graphical print out of the curve should be included in the data for all quadratic curves to demonstrate that it is a good fit and has been reviewed for "fit".

4) The analyst will routinely run six standards for their calibration.

   - All six may be used for the average response factor curve (5 required).
   - All six must be used for the quadratic curve.
   - The lowest standard must be less than or equal to the project RL.

Initial Calibration Criteria

1) All CCCs must be ≤ 30% RSD in order for the curve to be acceptable and the CCC's may use an average response factor curve. Where the term target compound is used below it refers to non-CCC's

2) Where a target compound is ≤ 15% RSD an average response factor curve may be used.

3) Where a target compound is ≥ 15% but ≤ 30% the analyst will review the curve techniques to select a "best fit" curve. An average response factor curve may be used if

the analyst shows that the average response factor is an acceptable fit in the range that the curve is being used. A graphical representation of the curve should be presented for documentation. If the quadratic is clearly a better fit it must be used.

4) Where a quadratic or polynomial curve is used R must be ≥ .995

5) Compound list will be divided into two lists: list one (reliable performers) and list two (poor performers). **List one compounds should always have a %RSD less than 30 percent or correlation coefficient of .995 with an allowance for up to two sporadic marginal failures for volatiles and four for semivolatiles.** Sporadic marginal failures for these compounds should be </= 40% or .990. Sporadic marginal failures require a print out of the curve.

6) List two compounds are comprised of the list of known poor performers. **List two analytes may use an average response factor curve, where the %RSD is ≤ 15% and where the %RSDs > 15% and ≤ 60% a "best fit" curve will be selected. For these compounds (%RSD > 15%) a print out of the curve will be provided as a graphical documentation of curve performance and of "best-fit" selection**.

7) Documentation: Raw target curve summary with all compounds set to average response factor will be provided. If quadratic or polynomial equations are used a reprint of the curve table will be provided to show the correlation coefficient for the "best fit" equations. And as noted above, compounds that need additional documentation to demonstrate the curve fit will have a graphical presentation of the curve provided for reference.

8) Any analyte not on list one or list two would be held to specific criteria based on project specific requirements.

Minimum RRF Criteria

1) SPCCs must have an RRF ≥ 0.050

2) All other target compounds must have an RRF of ≥ 0.010,

Continuing Calibration Verification

The continuing calibration verification requirements for DoD work are listed in SOP PT-QA-025.

Calculation Type

1) Average Response factor curves should be verified using a %Difference Equation. The %Difference Equation compares the RRF factor calculated for the Calibration Verification Standard to the Average RRF of the Curve.

2) The Quadratic Curves should be verified using a %Drift Equation. The %Drift Equation compares the measured value of the Calibration Verification Standard to the theoretical value of the standard.

% Diff. & % Drift Criteria

1) CCCs must be ≤ 20% Diff.

2) List 1 compounds that are Non CCC's must be ≤ 25% Diff or Drift

3) Up to 2 Volatile and 4 Semivolatile compounds that are List 1 analytes may exceed the 25% criteria but must be ≤ 40%.

4) List 2 Target Analytes including Appendix IX compounds will be accepted where the % difference or % Drift ≤ 50%.

5) Where a CCV is out high by > 50% and the compound is ND in the samples, the samples may be reported with narration.

RRF Criteria

1) SPCCs must be ≥ 0.05

2) All other compounds must be ≥ 0.01

Narrative Issues:

1) All %RSD that > 30% must be narrated.

2) All % D or Drift > 25% must be narrated.

3) Any other criteria exceedances aside from these should be narrated

4) Using an average response factor curve for a % RSD ≥ 30% must be narrated.

5) If a list 1 compound is not found in the sample, up to 50% D or Drift may be accepted with narration subject to determination that it is acceptable for the specific project.

6) If a list 2 compound is > 50% D or Drift (out high) and it is not found in the samples it may be reported with narration.

Note: These criterions are subject to project specific criteria, which may vary, depending on project compounds of concern and the usability needs of the project.

COMPOUND	SW846	LIST	QC TYPE
2,4,6-Trichlorophenol	8270C	**CCC**	**CCC**
2,4-Dichlorophenol	8270C	**CCC**	**CCC**
2-Nitrophenol	8270C	**CCC**	**CCC**
4-Chloro-3-methylphenol	8270C	**CCC**	**CCC**
Acenaphthene	8270C	**CCC**	**CCC**
Benzo(a)pyrene	8270C	**CCC**	**CCC**
Fluoranthene	8270C	**CCC**	**CCC**
Pentachlorophenol	8270C	**CCC**	**CCC**
Phenol	8270C	**CCC**	**CCC**
Di-n-octyl phthalate	8270C	**CCC**	**CCC**
Hexachlorobutadiene	8270C	**CCC**	**CCC**
N-Nitrosodiphenylamine	8270C	**CCC**	**CCC**
1,4-Dichlorobenzene	8270C	**CCC**	**CCC**
2,4,5-Trichlorophenol	8270C	1	
2,4-Dimethylphenol	8270C	1	
2,4-Dinitrotoluene	8270C	1	
2,6-Dinitrotoluene	8270C	1	
2-Chloronaphthalene	8270C	1	
2-Chlorophenol	8270C	1	
2-Methylnaphthalene	8270C	1	
2-Methylphenol	8270C	1	
4-Bromophenyl phenyl ether	8270C	1	
4-Chlorophenyl phenyl ether	8270C	1	
4-Methylphenol	8270C	1	
Acenaphthylene	8270C	1	
Anthracene	8270C	1	
Benzo(a)anthracene	8270C	1	
Benzo(b)fluoranthene	8270C	1	
Benzo(k)fluoranthene	8270C	1	
bis(2-Chloroethoxy)methane	8270C	1	
bis(2-Chloroethyl) ether	8270C	1	
Chrysene	8270C	1	

COMPOUND	SW846	LIST	QC TYPE
Dibenzofuran	8270C	1	
Fluorene	8270C	1	
Hexachlorobenzene	8270C	1	
Hexachloroethane	8270C	1	
Isophorone	8270C	1	
Naphthalene	8270C	1	
Nitrobenzene	8270C	1	
N-Nitrosodi-n-propylamine	8270C	**1**	**SPCC**
Phenanthrene	8270C	1	
Pyrene	8270C	1	
3&4 Methylphenol total	8270C	1	
1,2,4-Trichlorobenzene	8270C	1	
Benzo(ghi)perylene	8270C	1	
2,4-Dinitrophenol	8270C	**2**	**SPCC**
2-Nitroaniline	8270C	2	
3,3'-Dichlorobenzidine	8270C	2	
3-Nitroaniline	8270C	2	
4,6-Dinitro-2-methylphenol	8270C	2	
4-Chloroaniline	8270C	2	
4-Nitroaniline	8270C	2	
4-Nitrophenol	8270C	**2**	**SPCC**
bis(2-Ethylhexyl) phthalate	8270C	2	
Butyl benzyl phthalate	8270C	2	
Carbazole	8270C	2	
Dibenz(a,h)anthracene	8270C	2	
Diethyl phthalate	8270C	2	
Dimethyl phthalate	8270C	2	
Di-n-butyl phthalate	8270C	2	
Hexachlorocyclopentadiene	8270C	**2**	**SPCC**
Indeno(1,2,3-cd)pyrene	8270C	2	
1,2,4,5-Tetrachlorobenzene	8270C	2	
1,2-Dichlorobenzene	8270C	2	
1,2-Diphenylhydrazine	8270C	2	
1,3,5-Trinitrobenzene	8270C	2	
1,3-Dichlorobenzene	8270C	2	
1,3-Dinitrobenzene	8270C	2	
1,4-Dioxane	8270C	2	

COMPOUND	SW846	LIST	QC TYPE
1,4-Naphthoquinone	8270C	2	
1-Methylnaphthalene	8270C	2	
1-Naphthylamine	8270C	2	
2,2'-oxybis(1-Chloropropane)	8270C	2	
2,3,4,6-Tetrachlorophenol	8270C	2	
2,3,5,6-Tetrachlorophenol	8270C	2	
2,6-Dichlorophenol	8270C	2	
2-Acetylaminofluorene	8270C	2	
2-Methyl-4,6-dinitrophenol	8270C	2	
2-Naphthylamine	8270C	2	
2-Picoline	8270C	2	
2-sec-Butyl-4,6-dinitrophenol	8270C	2	
3,3'-Dimethylbenzidine	8270C	2	
3-Methylcholanthrene	8270C	2	
4,4'-Methylenebis(2-chloroaniline)	8270C	2	
4,6-Dinitro-o-cresol	8270C	2	
4-Aminobiphenyl	8270C	2	
4-Nitroquinoline-1-oxide	8270C	2	
5-Nitro-o-toluidine	8270C	2	
6-Methylchrysene	8270C	2	
7,12-Dimethylbenz(a)anthracene	8270C	2	
a,a-Dimethylphenethylamine	8270C	2	
alpha,alpha-Dimethylphenethylamine	8270C	2	
Aniline	8270C	2	
Aramite	8270C	2	
Aramite (total)	8270C	2	
Benzenethiol	8270C	2	
Benzidine	8270C	2	
bis(2-Chloroisopropyl) ether	8270C	2	
Chlorobenzilate	8270C	2	
Cresols (total)	8270C	2	
Diallate	8270C	2	
Dibenz(a,h)acridine	8270C	2	
Dibenzo(a,h)anthracene	8270C	2	
Dimethoate	8270C	2	
Dinoseb	8270C	2	
Disulfoton	8270C	2	

COMPOUND	SW846	LIST	QC TYPE
Ethyl methanesulfonate	8270C	2	
Famphur	8270C	2	
Hexachloropropene	8270C	2	
Isodrin	8270C	2	
Isosafrole	8270C	2	
Kepone	8270C	2	
m-Dinitrobenzene	8270C	2	
Methapyrilene	8270C	2	
Methyl methanesulfonate	8270C	2	
Methyl parathion	8270C	2	
N-Nitrosodiethylamine	8270C	2	
N-Nitrosodimethylamine	8270C	2	
N-Nitrosodi-n-butylamine	8270C	2	
N-Nitrosomethylethylamine	8270C	2	
N-Nitrosomorpholine	8270C	2	
N-Nitrosopiperidine	8270C	2	
N-Nitrosopyrrolidine	8270C	2	
O,O,O-Triethyl phosphorothioate	8270C	2	
o-Toluidine	8270C	2	
Parathion	8270C	2	
p-Chloroaniline	8270C	2	
p-Chlorobenzilate	8270C	2	
p-Chloro-m-cresol	8270C	2	
p-Dimethylaminoazobenzene	8270C	2	
Pentachlorobenzene	8270C	2	
Pentachloroethane	8270C	2	
Pentachloronitrobenzene	8270C	2	
Phenacetin	8270C	2	
Phorate	8270C	2	
p-Nitroaniline	8270C	2	
p-Phenylene diamine	8270C	2	
Pronamide	8270C	2	
Pyridine	8270C	2	
Safrole	8270C	2	
Sulfotepp	8270C	2	
Thionazin	8270C	2	
1,1'-Biphenyl	8270C	*	

COMPOUND	SW846	LIST	QC TYPE
Acetophenone	8270C	*	
Atrazine	8270C	*	
Benzaldehyde	8270C	*	
Caprolactam	8270C	*	
Benzoic acid	8270C	*	
Benzyl alcohol	8270C	*	
Indene	8270C	*	
Quindine	8270C	*	
1,4-Oxathiane	8270C	*	
Dimethyl Disulfide	8270C	*	
p-chlorophenyl metyl sulfide	8270C	*	
p-chlorophenyl metyl sulfone	8270C	*	
p-chloropheyny methyl sulfoxide	8270C	*	
Hexachlorophene	8270C	TIC	

* SPECIFIC CRITERIA WOULD BE IMPLEMENTED ON A PROJECT SPECIFIC BASIS WHEN REQUIRED.

Tables

Table 1
TestAmerica Pittsburgh Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)[2]	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330

Table 1
TestAmerica Pittsburgh Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
4-Phenylenediamine	106-50-3	200	6600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butyl phthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330
Atrazine	1912-24-9	10	330

Table 1
TestAmerica Pittsburgh Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
1,4-Dioxane	123-91-1	10	330
Benzaldehyde	100-52-7	10	330
Acetophenone	98-68-2	10	330
Caprolactam	105-60-2	10	330
1,1-Biphenyl	92-52-4	10	330
2-Naphthylamine	91-59-8	10	330

[1] The TestAmerica Pittsburgh primary standard is the standard normally used at TestAmerica Pittsburgh. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.

2 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

Table 2

TestAmerica Pittsburgh Appendix IX[1] Routine Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
Acetophenone	98-86-2	10	330
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	20	660
3-Methylphenol	108-39-4	10	330
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	3300
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	20	660
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	10	330
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin[2]	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp[2]	3689-24-5	50	1600
Phorate[2]	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate[3]	2303-16-4	20	660
Dimethoate[2]	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600

Table 2

TestAmerica Pittsburgh Appendix IX[1] Routine Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pronamide	23950-58-5	20	660
Disulfoton[2]	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	20	660
Methyl Parathion[2]	298-00-0	10	330
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion[2]	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	50	1600
Isodrin[3]	465-73-6	10	330
Kepone	143-50-0	40	1300
Famphur[2]	52-85-7	100	3300
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate[3]	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	50	1600
2-Acetylaminofluorene	53-96-3	20	660
Dibenz(a,j)acridine	224-42-0	20	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	50	1600

[1] The Appendix IX standard contains additional analytes required for the Appendix IX list. The TestAmerica Pittsburgh primary standard must also be analyzed to include all of the Appendix IX list.

[2] May also be analyzed by method 8141A, which can achieve lower reporting limits.

[3] May also be analyzed by method 8081A, which can achieve lower reporting limits

Table 2 A 8270C Water Low Level Method Code 42 Reporting Limits	
Compound	**Reporting Limit ug/L**
a,a-Dimethylphenethylamine	1
Acenaphthene	0.2
Acenaphthylene	0.2
Acetophenone	1
2-Acetylaminofluorene	1
4-Aminobiphenyl	1
Aniline	1
Anthracene	0.2
Aramite	1
Aramite (total)	1
Atrazine	1
Benzaldehyde	1
Benzenethiol	1
Benzidine	20
Benzo(a)anthracene	0.2
Benzo(b)fluoranthene	0.2
Benzo(k)fluoranthene	0.2
Benzoic acid	5
Benzo(ghi)perylene	0.2
Benzo(a)pyrene	0.2
Benzotrichloride	10
Benzyl alcohol	1
1,1'-Biphenyl	1
Biphenyl	1
bis(2-Chloroethoxy)methane	1
bis(2-Chloroethyl) ether	0.2
bis(2-Chloroisopropyl) ether	0.2
bis(2-Ethylhexyl) phthalate	1
Bis(4-hydroxyphenyl)methane	1
Bis(2-hydroxyphenyl)methane	1
2-Bromonaphthalene	1
4-Bromophenyl phenyl ether	1
Butyl benzyl phthalate	1
Caprolactam	1

Table 2 A 8270C Water Low Level Method Code 42 Reporting Limits	
Compound	**Reporting Limit ug/L**
Carbaryl	1
Carbazole	0.2
p-Chloroaniline	1
4-Chloroaniline	1
Chlorobenzilate	1
p-Chlorobenzilate	1
4-Chloro-3-methylphenol	1
p-Chloro-m-cresol	1
2-Chloronaphthalene	0.2
2-Chlorophenol	1
4-Chlorophenyl phenyl ether	1
Chrysene	0.2
6-Methylchrysene	1
Cresols (total)	1
Diallate	1
Dibenz(a,h)acridine	1
Dibenz(a,h)anthracene	0.2
Dibenzo(a,h)anthracene	0.2
Dibenzofuran	1
1,2-Dibromo-3-chloropropane	1
Di-n-butyl phthalate	1
1,2-Dichlorobenzene	0.2
o-Dichlorobenzene	0.2
1,3-Dichlorobenzene	0.2
m-Dichlorobenzene	0.2
1,4-Dichlorobenzene	0.2
p-Dichlorobenzene	0.2
3,3'-Dichlorobenzidine	1
2,3-Dichlorophenol	1
2,4-Dichlorophenol	0.2
2,6-Dichlorophenol	0.2
2,5-Dichlorophenol	1
Diethyl phthalate	1
Dimethoate	1
p-Dimethylaminoazobenzene	1

Table 2 A 8270C Water Low Level Method Code 42 Reporting Limits	
Compound	**Reporting Limit ug/L**
N,N-Dimethylaniline	1
7,12-Dimethylbenz(a)anthracene	1
3,3'-Dimethylbenzidine	10
alpha,alpha-Dimethylphenethylamine	1
2,4-Dimethylphenol	1
Dimethyl phthalate	1
m-Dinitrobenzene	1
1,3-Dinitrobenzene	1
2-Methyl-4,6-dinitrophenol	5
4,6-Dinitro-o-cresol	5
4,6-Dinitro-2-methylphenol	5
2,4-Dinitrophenol	5
2,4-Dinitrotoluene	1
2,6-Dinitrotoluene	1
2-sec-Butyl-4,6-dinitrophenol	1
Dinoseb	1
Di-n-octyl phthalate	1
1,4-Dioxane	0.2
1,2-Diphenylhydrazine	0.2
1,2-Diphenylhydrazine (as Azobenzene)	0.2
Disulfoton	1
Ethyl methanesulfonate	1
Famphur	10
Fluoranthene	0.2
Fluorene	0.2
Hexachlorobenzene	0.2
Hexachlorobutadiene	0.2
Hexachlorocyclopentadiene	1
Hexachloroethane	1
Hexachlorophene	--
Hexachloropropene	1
Indeno(1,2,3-cd)pyrene	0.2
Isodrin	1
Isophorone	1
Isosafrole	1

Table 2 A 8270C Water Low Level Method Code 42 Reporting Limits	
Compound	**Reporting Limit ug/L**
Kepone	4
Methapyrilene	1
3-Methylcholanthrene	1
4,4'-Methylenebis(2-chloroaniline)	1
Methyl methanesulfonate	1
2-Methylnaphthalene	0.2
1-Methylnaphthalene	0.2
Methyl parathion	1
2-Methylphenol	1
3-Methylphenol	1
4-Methylphenol	1
3-Methylphenol & 4-Methylphenol	1
Naphthalene	0.2
1,4-Naphthoquinone	1
1-Naphthylamine	1
2-Naphthylamine	1
2-Nitroaniline	5
3-Nitroaniline	5
4-Nitroaniline	5
p-Nitroaniline	5
Nitrobenzene	0.2
4-Nitrobiphenyl	1
2-Nitrophenol	1
4-Nitrophenol	5
4-Nitroquinoline-1-oxide	10
N-Nitrosodi-n-butylamine	1
N-Nitrosodiethylamine	1
N-Nitrosodimethylamine	1
N-Nitrosodiphenylamine	0.2
N-Nitrosodiphenylamine (1)	0.2
N-Nitrosodi-n-propylamine	0.2
N-Nitrosomethylethylamine	1
N-Nitrosomorpholine	1
N-Nitrosopiperidine	1
N-Nitrosopyrrolidine	1

Table 2 A 8270C Water Low Level Method Code 42 Reporting Limits	
Compound	**Reporting Limit ug/L**
5-Nitro-o-toluidine	10
Octachlorocyclopentene	1
Octachlorostyrene	1
2,2'-oxybis(1-Chloropropane)	0.2
Parathion	1
Pentachlorobenzene	1
Pentachloroethane	2
Pentachloronitrobenzene	1
Pentachlorophenol	1
Phenacetin	1
Phenanthrene	0.2
Phenol	0.2
p-Phenylene diamine	40
Phorate	1
2-Picoline	1
Pronamide	1
Pyrene	0.2
Pyridine	1
Safrole	1
Sevin	1
Sulfotepp	1
1,2,4,5-Tetrachlorobenzene	1
2,3,4,6-Tetrachlorophenol	1
2,3,5,6-Tetrachlorophenol	1
1,2,3,4-Tetrahydronaphthalene	1
Thionazin	1
o-Toluidine	1
1,2,4-Trichlorobenzene	0.2
2,4,5-Trichlorophenol	1
2,4,6-Trichlorophenol	1
O,O,O-Triethyl phosphorothioate	1
Trifluralin	1
1,3,5-Trinitrobenzene	1
1-Nitronaphthalene	1
1,2,3,4-Tetrachlorobenzene	1

Table 2 A **8270C Water Low Level Method Code 42 Reporting Limits**	
Compound	**Reporting Limit ug/L**
3&4 Methylphenol total	1

Table 2 B 8270C Soil Low Level Method Code 42 Reporting Limits	
Compuound	**Reporting Limit ug/kg**
a,a-Dimethylphenethylamine	33
Acenaphthene	6.7
Acenaphthylene	6.7
Acetophenone	33
2-Acetylaminofluorene	33
4-Aminobiphenyl	33
Aniline	33
Anthracene	6.7
Aramite	33
Aramite (total)	33
Atrazine	33
Benzaldehyde	33
Benzenethiol	330
Benzidine	670
Benzo(a)anthracene	6.7
Benzo(b)fluoranthene	6.7
Benzo(k)fluoranthene	6.7
Benzoic acid	170
Benzo(ghi)perylene	6.7
Benzo(a)pyrene	6.7
Benzyl alcohol	33
1,1'-Biphenyl	33
bis(2-Chloroethoxy)methane	33
bis(2-Chloroethyl) ether	6.7
bis(2-Chloroisopropyl) ether	6.7
bis(2-Ethylhexyl) phthalate	33
4-Bromophenyl phenyl ether	33
Butyl benzyl phthalate	33
Caprolactam	33
Carbazole	6.7
4-Chloroaniline	33
Chlorobenzilate	33
4-Chloro-3-methylphenol	33
2-Chloronaphthalene	6.7

Table 2 B 8270C Soil Low Level Method Code 42 Reporting Limits	
Compuound	**Reporting Limit ug/kg**
2-Chlorophenol	33
4-Chlorophenyl phenyl ether	33
Chrysene	6.7
6-Methylchrysene	33
Diallate	33
Dibenz(a,h)acridine	33
Dibenz(a,h)anthracene	6.7
Dibenzo(a,h)anthracene	6.7
Dibenzofuran	33
Di-n-butyl phthalate	33
1,2-Dichlorobenzene	6.7
1,3-Dichlorobenzene	6.7
1,4-Dichlorobenzene	6.7
3,3'-Dichlorobenzidine	33
2,4-Dichlorophenol	6.7
2,6-Dichlorophenol	6.7
Diethyl phthalate	33
O,O-Diethyl-O-(2-pyrazinyl) phosphorothioate	33
Dimethoate	33
p-Dimethylaminoazobenzene	33
7,12-Dimethylbenz(a)anthracene	33
3,3'-Dimethylbenzidine	170
alpha,alpha-Dimethylphenethylamine	33
2,4-Dimethylphenol	33
Dimethyl phthalate	33
m-Dinitrobenzene	33
1,3-Dinitrobenzene	33
4,6-Dinitro-2-methylphenol	170
2,4-Dinitrophenol	170
2,4-Dinitrotoluene	33
2,6-Dinitrotoluene	33
Dinoseb	33
Di-n-octyl phthalate	33
1,4-Dioxane	6.7
1,2-Diphenylhydrazine	6.7

Table 2 B 8270C Soil Low Level Method Code 42 Reporting Limits	
Compuound	**Reporting Limit ug/kg**
Disulfoton	33
Ethyl methanesulfonate	33
Famphur	330
Fluoranthene	6.7
Fluorene	6.7
Hexachlorobenzene	6.7
Hexachlorobutadiene	6.7
Hexachlorocyclopentadiene	33
Hexachloro-1,3-cyclopentadiene	33
Hexachloroethane	33
Hexachlorophene	--
Hexachloropropene	33
Indeno(1,2,3-cd)pyrene	6.7
Isodrin	33
Isophorone	33
Isosafrole	33
Kepone	1300
Methapyrilene	33
3-Methylcholanthrene	33
4,4'-Methylenebis(2-chloroaniline)	33
Methyl methanesulfonate	33
2-Methylnaphthalene	6.7
1-Methylnaphthalene	6.7
Methyl parathion	33
2-Methylphenol	33
4-Methylphenol	33
3-Methylphenol & 4-Methylphenol	33
Naphthalene	6.7
1,4-Naphthoquinone	33
1-Naphthylamine	33
2-Naphthylamine	33
2-Nitroaniline	170
3-Nitroaniline	170
4-Nitroaniline	170
Nitrobenzene	6.7

Table 2 B 8270C Soil Low Level Method Code 42 Reporting Limits	
Compuound	**Reporting Limit ug/kg**
2-Nitrophenol	33
4-Nitrophenol	170
4-Nitroquinoline-1-oxide	170
N-Nitrosodi-n-butylamine	33
N-Nitrosodiethylamine	33
N-Nitrosodimethylamine	33
N-Nitrosodiphenylamine (1)	6.7
N-Nitrosodiphenylamine	6.7
N-Nitrosodi-n-propylamine	6.7
N-Nitrosomethylethylamine	33
N-Nitrosomorpholine	33
N-Nitrosopiperidine	33
N-Nitrosopyrrolidine	33
N-Nitro-o-toluidine	33
5-Nitro-o-toluidine	33
2,2'-oxybis(1-Chloropropane)	6.7
Parathion	33
Pentachlorobenzene	33
Pentachloroethane	33
Pentachloronitrobenzene	33
Pentachlorophenol	33
Phenacetin	33
Phenanthrene	6.7
Phenol	6.7
p-Phenylene diamine	670
Phorate	33
2-Picoline	33
Pronamide	33
Pyrene	6.7
Pyridine	33
Safrole	33
Sulfotepp	33
1,2,4,5-Tetrachlorobenzene	33
2,3,4,6-Tetrachlorophenol	33
2,3,5,6-Tetrachlorophenol	33

Table 2 B 8270C Soil Low Level Method Code 42 Reporting Limits	
Compuound	**Reporting Limit ug/kg**
Tetraethyldithiopyrophosphate	33
Thionazin	33
1,2,4-Trichlorobenzene	6.7
2,4,5-Trichlorophenol	33
2,4,6-Trichlorophenol	33
O,O,O-Triethyl phosphorothioate	33
1,3,5-Trinitrobenzene	33

Table 2 C 8270C Low Level Water Method Code QL Reporting Limits	
Compound	**Reporting Limit ug/L**
a,a-Dimethylphenethylamine	10
Acenaphthene	2
Acenaphthylene	2
Acetophenone	10
2-Acetylaminofluorene	10
4-Aminobiphenyl	10
Aniline	10
Anthracene	2
Aramite	10
Aramite (total)	10
Atrazine	10
Benzaldehyde	10
Benzenethiol	10
Benzidine	200
Benzo(a)anthracene	2
Benzo(b)fluoranthene	2
Benzo(k)fluoranthene	2
Benzoic acid	50
Benzo(ghi)perylene	2
Benzo(a)pyrene	2
Benzotrichloride	100
Benzyl alcohol	10

Table 2 C 8270C Low Level Water Method Code QL Reporting Limits	
Compound	**Reporting Limit ug/L**
1,1'-Biphenyl	10
Biphenyl	10
bis(2-Chloroethoxy)methane	10
bis(2-Chloroethyl) ether	2
bis(2-Chloroisopropyl) ether	2
bis(2-Ethylhexyl) phthalate	10
2-Bromonaphthalene	10
4-Bromophenyl phenyl ether	10
Butyl benzyl phthalate	10
Caprolactam	10
Carbaryl	10
Carbazole	2
p-Chloroaniline	10
4-Chloroaniline	10
Chlorobenzilate	10
p-Chlorobenzilate	10
4-Chloro-3-methylphenol	10
p-Chloro-m-cresol	10
2-Chloronaphthalene	2
2-Chlorophenol	10
4-Chlorophenyl phenyl ether	10
Chrysene	2
6-Methylchrysene	10
Cresols (total)	10
Diallate	10
Dibenz(a,h)acridine	10
Dibenz(a,h)anthracene	2
Dibenzo(a,h)anthracene	2
Dibenzofuran	10
1,2-Dibromo-3-chloropropane	10
Di-n-butyl phthalate	10
1,2-Dichlorobenzene	2
o-Dichlorobenzene	2
1,3-Dichlorobenzene	2
m-Dichlorobenzene	2

Table 2 C 8270C Low Level Water Method Code QL Reporting Limits	
Compound	**Reporting Limit ug/L**
1,4-Dichlorobenzene	2
p-Dichlorobenzene	2
3,3'-Dichlorobenzidine	10
2,4-Dichlorophenol	2
2,6-Dichlorophenol	2
Diethyl phthalate	10
Dimethoate	10
p-Dimethylaminoazobenzene	10
N,N-Dimethylaniline	10
7,12-Dimethylbenz(a)anthracene	10
3,3'-Dimethylbenzidine	100
alpha,alpha-Dimethylphenethylamine	10
2,4-Dimethylphenol	10
Dimethyl phthalate	10
m-Dinitrobenzene	10
1,3-Dinitrobenzene	10
2-Methyl-4,6-dinitrophenol	50
4,6-Dinitro-o-cresol	50
4,6-Dinitro-2-methylphenol	50
2,4-Dinitrophenol	50
2,4-Dinitrotoluene	10
2,6-Dinitrotoluene	10
2-sec-Butyl-4,6-dinitrophenol	10
Dinoseb	10
Di-n-octyl phthalate	10
1,4-Dioxane	2
Diphenylamine	2
1,2-Diphenylhydrazine	2
1,2-Diphenylhydrazine (as Azobenzene)	2
Disulfoton	10
Ethyl methanesulfonate	10
Famphur	100
Fluoranthene	2
Fluorene	2
Hexachlorobenzene	2

Table 2 C 8270C Low Level Water Method Code QL Reporting Limits	
Compound	**Reporting Limit ug/L**
Hexachlorobutadiene	2
Hexachlorocyclopentadiene	10
Hexachloroethane	10
Hexachlorophene	--
Hexachloropropene	10
Indeno(1,2,3-cd)pyrene	2
Isodrin	10
Isophorone	10
Isosafrole	10
Kepone	40
Methapyrilene	10
3-Methylcholanthrene	10
4,4'-Methylenebis(2-chloroaniline)	10
Methyl methanesulfonate	10
2-Methylnaphthalene	2
1-Methylnaphthalene	2
Methyl parathion	10
2-Methylphenol	10
3-Methylphenol	10
4-Methylphenol	10
3-Methylphenol & 4-Methylphenol	10
Naphthalene	2
1,4-Naphthoquinone	10
1-Naphthylamine	10
2-Naphthylamine	10
2-Nitroaniline	50
3-Nitroaniline	50
4-Nitroaniline	50
p-Nitroaniline	50
Nitrobenzene	2
4-Nitrobiphenyl	10
2-Nitrophenol	10
4-Nitrophenol	50
4-Nitroquinoline-1-oxide	100
N-Nitrosodi-n-butylamine	10

Table 2 C 8270C Low Level Water Method Code QL Reporting Limits	
Compound	**Reporting Limit ug/L**
N-Nitrosodiethylamine	10
N-Nitrosodimethylamine	10
N-Nitrosodiphenylamine	2
N-Nitrosodiphenylamine (1)	2
N-Nitrosodi-n-propylamine	2
N-Nitrosomethylethylamine	10
N-Nitrosomorpholine	10
N-Nitrosopiperidine	10
N-Nitrosopyrrolidine	10
5-Nitro-o-toluidine	100
2,2'-oxybis(1-Chloropropane)	2
Parathion	10
Pentachlorobenzene	10
Pentachloroethane	20
Pentachloronitrobenzene	10
Pentachlorophenol	10
Phenacetin	10
Phenanthrene	2
Phenol	2
p-Phenylene diamine	400
Phorate	10
2-Picoline	10
Pronamide	10
Pyrene	2
Pyridine	10
Safrole	10
Sevin	10
Sulfotepp	10
1,2,4,5-Tetrachlorobenzene	10
2,3,4,6-Tetrachlorophenol	10
2,3,5,6-Tetrachlorophenol	10
1,2,3,4-Tetrahydronaphthalene	10
Thionazin	10
o-Toluidine	10
1,2,4-Trichlorobenzene	2

Table 2 C 8270C Low Level Water Method Code QL Reporting Limits	
Compound	**Reporting Limit ug/L**
2,4,5-Trichlorophenol	10
2,4,6-Trichlorophenol	10
O,O,O-Triethyl phosphorothioate	10
Trifluralin	10
1,3,5-Trinitrobenzene	10
1-Nitronaphthalene	10
3&4 Methylphenol total	10

Table 2 D 8270C Low Level Soil Method Code QL Reporting Limits	
Compound	**Reporting Limit ug/L**
a,a-Dimethylphenethylamine	330
Acenaphthene	67
Acenaphthylene	67
Acetophenone	330
2-Acetylaminofluorene	330
4-Aminobiphenyl	330
Aniline	330
Anthracene	67
Aramite	330
Aramite (total)	330
Atrazine	330
Benzaldehyde	330
Benzenethiol	330
Benzidine	6700
Benzo(a)anthracene	67
Benzo(b)fluoranthene	67
Benzo(k)fluoranthene	67
Benzoic acid	1700
Benzo(ghi)perylene	67
Benzo(a)pyrene	67
Benzyl alcohol	330
1,1'-Biphenyl	330
bis(2-Chloroethoxy)methane	330
bis(2-Chloroethyl) ether	67
bis(2-Chloroisopropyl) ether	67
bis(2-Ethylhexyl) phthalate	330
4-Bromophenyl phenyl ether	330
Butyl benzyl phthalate	330
Caprolactam	330
Carbazole	67
p-Chloroaniline	330
4-Chloroaniline	330
Chlorobenzilate	330
p-Chlorobenzilate	330
4-Chloro-3-methylphenol	330

Table 2 D 8270C Low Level Soil Method Code QL Reporting Limits	
Compound	**Reporting Limit ug/L**
p-Chloro-m-cresol	330
2-Chloronaphthalene	67
2-Chlorophenol	330
4-Chlorophenyl phenyl ether	330
Chrysene	67
6-Methylchrysene	330
Diallate	330
Dibenz(a,h)acridine	330
Dibenz(a,h)anthracene	67
Dibenzo(a,h)anthracene	67
Dibenzofuran	330
Di-n-butyl phthalate	330
1,2-Dichlorobenzene	67
o-Dichlorobenzene	67
1,3-Dichlorobenzene	67
m-Dichlorobenzene	67
1,4-Dichlorobenzene	67
p-Dichlorobenzene	67
3,3'-Dichlorobenzidine	330
2,4-Dichlorophenol	67
2,6-Dichlorophenol	67
Diethyl phthalate	330
O,O-Diethyl-O-(2-pyrazinyl) phosphorothioate	330
Dimethoate	330
p-Dimethylaminoazobenzene	330
7,12-Dimethylbenz(a)anthracene	330
3,3'-Dimethylbenzidine	1700
alpha,alpha-Dimethylphenethylamine	330
2,4-Dimethylphenol	330
Dimethyl phthalate	330
m-Dinitrobenzene	330
1,3-Dinitrobenzene	330
4,6-Dinitro-o-cresol	1700
2-Methyl-4,6-dinitrophenol	1700
4,6-Dinitro-2-methylphenol	1700

Table 2 D 8270C Low Level Soil Method Code QL Reporting Limits	
Compound	**Reporting Limit ug/L**
2,4-Dinitrophenol	1700
2,4-Dinitrotoluene	330
2,6-Dinitrotoluene	330
2-sec-Butyl-4,6-dinitrophenol	330
Dinoseb	330
Di-n-octyl phthalate	330
1,4-Dioxane	67
1,2-Diphenylhydrazine (as Azobenzene)	67
1,2-Diphenylhydrazine	67
Disulfoton	330
Ethyl methanesulfonate	330
Famphur	3300
Fluoranthene	67
Fluorene	67
Hexachlorobenzene	67
Hexachlorobutadiene	67
Hexachlorocyclopentadiene	330
Hexachloro-1,3-cyclopentadiene	330
Hexachloroethane	330
Hexachlorophene	--
Hexachloropropene	330
Indeno(1,2,3-cd)pyrene	67
Isodrin	330
Isophorone	330
Isosafrole	330
Kepone	13000
Methapyrilene	330
3-Methylcholanthrene	330
4,4'-Methylenebis(2-chloroaniline)	330
Methyl methanesulfonate	330
2-Methylnaphthalene	67
1-Methylnaphthalene	67
Methyl parathion	330
2-Methylphenol	330
4-Methylphenol	330

Table 2 D 8270C Low Level Soil Method Code QL Reporting Limits	
Compound	**Reporting Limit ug/L**
3-Methylphenol & 4-Methylphenol	330
Naphthalene	67
1,4-Naphthoquinone	330
1-Naphthylamine	330
2-Naphthylamine	330
2-Nitroaniline	1700
3-Nitroaniline	1700
m-Nitroaniline	1700
4-Nitroaniline	1700
p-Nitroaniline	1700
Nitrobenzene	67
2-Nitrophenol	330
4-Nitrophenol	1700
4-Nitroquinoline-1-oxide	1700
N-Nitrosodi-n-butylamine	330
N-Nitrosodiethylamine	330
N-Nitrosodimethylamine	330
N-Nitrosodiphenylamine (1)	67
N-Nitrosodiphenylamine	67
N-Nitrosodi-n-propylamine	67
N-Nitrosomethylethylamine	330
N-Nitrosomorpholine	330
N-Nitrosopiperidine	330
N-Nitrosopyrrolidine	330
5-Nitro-o-toluidine	330
2,2'-oxybis(1-Chloropropane)	67
Parathion	330
Pentachlorobenzene	330
Pentachloroethane	330
Pentachloronitrobenzene	330
Pentachlorophenol	330
Phenacetin	330
Phenanthrene	67
Phenol	67
p-Phenylene diamine	6700

Table 2 D **8270C Low Level Soil Method Code QL Reporting Limits**	
Compound	**Reporting Limit ug/L**
Phorate	330
2-Picoline	330
Pronamide	330
Pyrene	67
Pyridine	330
Safrole	330
Sulfotepp	330
1,2,4,5-Tetrachlorobenzene	330
2,3,4,6-Tetrachlorophenol	330
2,3,5,6-Tetrachlorophenol	330
Tetraethyldithiopyrophosphate	330
Thionazin	330
o-Toluidine	330
1,2,4-Trichlorobenzene	67
2,4,5-Trichlorophenol	330
2,4,6-Trichlorophenol	330
O,O,O-Triethyl phosphorothioate	330
1,3,5-Trinitrobenzene	330
3&4 Methylphenol total	330

Table 3

Reportable Analytes for TestAmerica Pittsburgh Standard Tests, Primary Standard

Analyte	CAS Number	Routinely Calibrated Compounds	TCLP	TCL	Appendix IX
Pyridine	110-86-1	X	X		X
N-nitrosodimethylamine	62-75-9	X			X
Aniline	62-53-3	X			X
Phenol	108-95-2	X		X	X
Bis(2-chloroethyl)ether	111-44-4	X		X	X
2-Chlorophenol	95-57-8	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X
Benzyl alcohol	100-51-6	X			X
1,2-Dichlorobenzene	95-50-1	X		X	X
2-Methylphenol	95-48-7	X	X	X	X
2,2'-oxybis(1-chloropropane)[1]	180-60-1	X		X	X
4-Methylphenol	106-44-5	X	X	X	X
N-Nitroso-di-n-propylamine	621-64-7	X		X	X
Hexachloroethane	67-72-1	X	X	X	X
Nitrobenzene	98-95-3	X	X	X	X
Isophorone	78-59-1	X		X	X
2-Nitrophenol	88-75-5	X		X	X
2,4-Dimethylphenol	105-67-9	X		X	X
Benzoic acid	65-85-0	X			
Bis(2-chloroethoxy)methane	111-91-1	X		X	X
2,4-Dichlorophenol	120-83-2	X		X	X
1,2,4-Trichlorobenzene	120-82-1	X		X	X
Naphthalene	91-20-3	X		X	X
4-Chloroaniline	106-47-8	X		X	X
Hexachlorobutadiene	87-68-3	X	X	X	X
4-Chloro-3-methylphenol	59-50-7	X		X	X
2-Methylnaphthalene	91-57-6	X		X	X
Hexachlorocyclopentadiene	77-47-4	X		X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X	X
2-Chloronaphthalene	91-58-7	X		X	X
2-Nitroaniline	88-74-4	X		X	X
Dimethyl phthalate	131-11-3	X		X	X
Acenaphthylene	208-96-8	X		X	X
3-Nitroaniline	99-09-2	X		X	X

Table 3

Reportable Analytes for TestAmerica Pittsburgh Standard Tests, Primary Standard

Analyte	CAS Number	Routinely Calibrated Compounds	TCLP	TCL	Appendix IX
Acenaphthene	83-32-9	X		X	X
2,4-Dinitrophenol	51-28-5	X		X	X
4-Nitrophenol	100-02-7	X		X	X
Dibenzofuran	132-64-9	X		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X		X	X
Diethylphthalate	84-66-2	X		X	X
4-Chlorophenyl phenyl ether	7005-72-3	X		X	X
Fluorene	86-73-7	X		X	X
4-Nitroaniline	100-01-6	X		X	X
4,6-Dinitro-2-methylphenol	534-52-1	X		X	X
N-Nitrosodiphenylamine	86-30-6	X		X	X
Azobenzene[4]	103-33-3	X			
4-Bromophenyl phenyl ether	101-55-3	X		X	X
Hexachlorobenzene	118-74-1	X	X	X	X
Pentachlorophenol	87-86-5	X	X	X	X
Phenanthrene	85-01-8	X		X	X
Anthracene	120-12-7	X		X	X
Carbazole	86-74-8	X		X	
Di-n-butyl phthalate	84-74-2	X		X	X
Fluoranthene	206-44-0	X		X	X
Benzidine	92-87-5				
Pyrene	129-00-0	X		X	X
Butyl benzyl phthalate	85-68-7	X		X	X
3,3'-Dichlorobenzidine	91-94-1	X		X	X
Benzo(a)anthracene	56-55-3	X		X	X
Bis(2-ethylhexyl)phthalate	117-81-7	X		X	X
Chrysene	218-01-9	X		X	X
Di-n-octylphthalate	117-84-0	X		X	X
Benzo(b)fluoranthene	205-99-2	X		X	X
Benzo(k)fluoranthene	207-08-9	X		X	X
Benzo(a)pyrene	50-32-8	X		X	X
Indeno(1,2,3-cd)pyrene	193-39-5	X		X	X
Dibenz(a,h)anthracene	53-70-3	X		X	X
Benzo(g,h,i)perylene	191-24-2	X		X	X
Atrazine	1912-24-9	X		X	
1,4-Dioxane	123-91-1	X		X	
Benzaldehyde	100-52-7	X		X	

Table 3

Reportable Analytes for TestAmerica Pittsburgh Standard Tests, Primary Standard

Analyte	CAS Number	Routinely Calibrated Compounds	TCLP	TCL	Appendix IX
Acetophenone	98-68-2	X		X	
Caprolactam	105-60-2	X		X	
1,1-Biphenyl	92-52-4	X		X	
2-Naphthylamine	91-59-8	X		X	

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether
[2] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4

Reportable analytes for TestAmerica Pittsburgh Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	Appendix IX
2-Picoline	109-06-8	X
N-Nitrosomethylethylamine	10595-95-6	X
Methyl methanesulfonate	66-27-3	X
N-Nitrosodiethylamine	55-18-5	X
Ethyl methanesulfonate	62-50-0	X
Pentachloroethane	76-01-7	X
Acetophenone	98-86-2	X
N-Nitrosopyrrolidine	930-55-2	X
N-Nitrosomorpholine	59-89-2	X
o-Toluidine	95-53-4	X
3-Methylphenol	108-39-4	X
N-Nitrosopiperidine	100-75-4	X
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1	X
a,a-Dimethyl-phenethylamine	122-09-8	X
2,6-Dichlorophenol	87-65-0	X
Hexachloropropene	1888-71-7	X
p-Phenylenediamine	106-50-3	X
n-Nitrosodi-n-butylamine	924-16-3	X
Safrole	94-59-7	X
1,2,4,5-Tetrachlorobenzene	95-94-3	X
Isosafrole	120-58-1	X
1,4-Dinitrobenzene	100-25-4	
1,4-Naphthoquinone	130-15-4	X
1,3-Dinitrobenzene	99-65-0	X
Pentachlorobenzene	608-93-5	X
1-Naphthylamine	134-32-7	X
2-Naphthylamine	91-59-8	X
2,3,4,6-Tetrachlorophenol	58-90-2	X
5-Nitro-o-toluidine	99-55-8	X
Thionazin[2]	297-97-2	X
1,3,5-Trinitrobenzene	99-35-4	X
Sulfotepp[2]	3689-24-5	X
Phorate[2]	298-02-2	X
Phenacetin	62-44-2	X
Diallate	2303-16-4	X
Dimethoate[2]	60-51-5	X
4-Aminobiphenyl	92-67-1	X
Pentachloronitrobenzene	82-68-8	X

Table 4

Reportable analytes for TestAmerica Pittsburgh Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	Appendix IX
Pronamide	23950-58-5	X
Disulfoton[2]	298-04-4	X
2-secbutyl-4,6-dinitrophenol (Dinoseb)[2]	88-85-7	X
Methyl parathion[2]	298-00-0	X
4-Nitroquinoline-1-oxide	56-57-5	X
Parathion[2]	56-38-2	X
Isodrin[3]	465-73-6	X
Kepone	143-50-0	X
Famphur[2]	52-85-7	X
Methapyrilene	91-80-5	X
Aramite	140-57-8	X
p-(Dimethylamino)azobenzene	60-11-7	X
p-Chlorobenzilate[3]	510-15-6	X
3,3'-Dimethylbenzidine	119-93-7	X
2-Acetylaminofluorene	53-96-3	X
Dibenz(a,j)acridine	224-42-0	
7,12-Dimethylbenz(a)anthracene	57-97-6	X
3-Methylcholanthrene	56-49-5	X
Hexachlorophene[4]	70-30-4	X
Diphenylamine[5]	122-39-4	X

[2] May also be analyzed by method 8141A, which can achieve lower reporting limits.

[3] May also be analyzed by method 8081A, which can achieve lower reporting limits

[4] Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram.

[5] Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

Table 5

Suggested Instrumental Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	40°C for 2 minutes
Column Temperature Program	40 - 320°C at 11.5°C/min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Total Run time	0.5 min based on the last compound of cont. Cal.
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, split / splitless
Sample Volume	1 or 2 µl
Carrier Gas	Helium at 30 cm/sec

Table 6

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 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d6 (Surrogate Standard)	99	42	71
Acetophenone	105	77	51
Aniline	93	66	
Benzaldehyde	77	105	106
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	111
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	111
Benzyl Alcohol	108	79	77
Caprolactam	113	55	56
1,2-Dichlorobenzene	146	148	111
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	121
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
1,1-Biphenyl	154	153	76
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Naphthylamine	143	115	116
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Atrazine	200	173	215
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	89	63
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	139	109	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	51	105
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	168
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	203
Benzidine	184	92	185
Pyrene	202	200	203
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Perylene-d12 (Internal Standard)	264	260	265
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	77
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	107
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	116
2-Naphthylamine	143	115	116
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	121	75	260
Phenacetin	108	179	109
Diallate	86	43	234
Dimethoate	87	93	125
4-Aminobiphenyl	169	168	170
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160
Parathion	109	97	291
Isodrin	193	66	195
Kepone	272	274	237
Famphur	218	125	93
Methapyrilene	58	97	72
Aramite 1	185	135	63
Aramite 2	185	135	63
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	213	211
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	277
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 9
8270C Routine LCS and Spike Control Compounds and Control Limits

Water			LCS			Spike		
Compound	**Units**	**Spike Level**	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
Acenaphthene	ug/L	50	40	97	32	31	131	39
4-Bromophenyl phenyl ether	ug/L	50	40	105	40	51	125	37
Butyl benzyl phthalate	ug/L	50	39	105	35	37	132	63
4-Chloro-3-methylphenol	ug/L	50	38	100	32	31	127	83
2-Chlorophenol	ug/L	50	38	97	31	10	129	139
1,4-Dichlorobenzene	ug/L	50	38	94	33	18	107	60
2,4-Dinitrotoluene	ug/L	50	37	103	32	41	130	53
Hexachloroethane	ug/L	50	35	96	43	10	111	63
4-Methylphenol	ug/L	100	33	106	34	28	125	77
Naphthalene	ug/L	50	38	98	39	29	118	45
4-Nitrophenol	ug/L	50	30	112	39	10	163	118
N-Nitrosodi-n-propylamine	ug/L	50	36	102	36	39	122	47
Pentachlorophenol	ug/L	50	13	120	56	10	165	98
Phenol	ug/L	50	36	98	35	10	135	115
Pyrene	ug/L	50	39	108	38	37	132	45
1,2,4-Trichlorobenzene	ug/L	50	39	97	32	10	142	52
Soil			**LCS**			**Spike**		
Compound	**Units**	**Spike Level**	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
Acenaphthene	ug/kg	1665	38	112	51	15	130	50
4-Bromophenyl phenyl ether	ug/kg	1665	46	120	81	27	136	48
Butyl benzyl phthalate	ug/kg	1665	47	115	54	27	130	48
4-Chloro-3-methylphenol	ug/kg	1665	39	111	52	16	128	52
2-Chlorophenol	ug/kg	1665	38	109	62	16	120	54
1,4-Dichlorobenzene	ug/kg	1665	36	107	57	20	105	62
2,4-Dinitrotoluene	ug/kg	1665	35	117	50	15	132	49
Hexachloroethane	ug/kg	1665	40	106	53	13	111	63
4-Methylphenol	ug/kg	3333	41	117	87	17	131	50
Naphthalene	ug/kg	1665	44	109	64	10	140	56
4-Nitrophenol	ug/kg	1665	30	125	43	10	154	88
N-Nitrosodi-n-propylamine	ug/kg	1665	36	114	45	30	118	51
Pentachlorophenol	ug/kg	1665	21	127	52	10	136	123
Phenol	ug/kg	1665	36	110	55	19	119	50
Pyrene	ug/kg	1665	43	118	48	10	168	69
1,2,4-Trichlorobenzene	ug/kg	1665	37	111	58	21	118	49

Table 9A
Low Level 8270C (Spike Method Code 42) - LCS and Spike Control Compounds and Control Limits

Water			LCS			Spike		
Compound	**Units**	**Spike Level**	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
Acenaphthene	ug/L	20	35	96	41	35	96	41
4-Bromophenyl phenyl ether	ug/L	20	39	94	40	39	94	40
Butyl benzyl phthalate	ug/L	20	33	106	40	33	106	40
4-Chloro-3-methylphenol	ug/L	20	41	99	42	41	99	42
2-Chlorophenol	ug/L	20	39	93	39	39	93	39
1,4-Dichlorobenzene	ug/L	20	36	91	41	36	91	41
2,4-Dinitrotoluene	ug/L	20	37	120	39	37	120	39
Hexachloroethane	ug/L	20	38	91	39	38	91	39
4-Methylphenol	ug/L	40	41	92	41	41	92	41
Naphthalene	ug/L	20	40	89	43	40	89	43
4-Nitrophenol	ug/L	20	39	110	42	39	110	42
N-Nitrosodi-n-propylamine	ug/L	20	41	96	43	41	96	43
Pentachlorophenol	ug/L	20	23	108	42	23	108	42
Phenol	ug/L	20	38	95	39	38	95	39
Pyrene	ug/L	20	30	106	42	30	106	42
1,2,4-Trichlorobenzene	ug/L	20	35	95	45	35	95	45
Soil			**LCS**			**Spike**		
Compound	**Units**	**Spike Level**	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
Acenaphthene	ug/kg	667	34	107	36	34	107	36
4-Bromophenyl phenyl ether	ug/kg	667	37	105	20	37	105	20
Butyl benzyl phthalate	ug/kg	667	35	110	34	35	110	34
4-Chloro-3-methylphenol	ug/kg	667	37	114	31	37	114	31
2-Chlorophenol	ug/kg	667	45	99	40	45	99	40
1,4-Dichlorobenzene	ug/kg	667	39	103	39	39	103	39
2,4-Dinitrotoluene	ug/kg	667	42	118	33	42	118	33
Hexachloroethane	ug/kg	667	40	102	37	40	102	37
4-Methylphenol	ug/kg	1334	40	113	42	40	113	42
Naphthalene	ug/kg	667	38	103	25	38	103	25
4-Nitrophenol	ug/kg	667	24	132	37	24	132	37
N-Nitrosodi-n-propylamine	ug/kg	667	39	111	32	39	111	32
Pentachlorophenol	ug/kg	667	18	117	37	18	117	37
Phenol	ug/kg	667	44	100	40	44	100	40
Pyrene	ug/kg	667	28	116	28	28	116	28
1,2,4-Trichlorobenzene	ug/kg	667	38	103	40	38	103	40
All samples are spiked with full analytes and the above compounds are the control analytes.								

Table 9B
Low Level 8270C (Spike Method Code QL)- LCS and Spike Control Compounds and Control Limits

Water			LCS			Spike		
Compound	**Units**	**Spike Level**	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
Acenaphthene	ug/L	200	40	97	32	31	131	39
4-Bromophenyl phenyl ether	ug/L	200	40	105	40	51	125	37
Butyl benzyl phthalate	ug/L	200	39	105	35	37	132	63
4-Chloro-3-methylphenol	ug/L	200	38	100	32	31	127	83
2-Chlorophenol	ug/L	200	38	97	31	10	129	139
1,4-Dichlorobenzene	ug/L	200	38	94	33	18	107	60
2,4-Dinitrotoluene	ug/L	200	37	103	32	41	130	53
Hexachloroethane	ug/L	200	35	96	43	10	111	63
4-Methylphenol	ug/L	400	33	106	34	28	125	77
Naphthalene	ug/L	200	38	98	39	29	118	45
4-Nitrophenol	ug/L	200	30	112	39	10	163	118
N-Nitrosodi-n-propylamine	ug/L	200	36	102	36	39	122	47
Pentachlorophenol	ug/L	200	13	120	56	10	165	98
Phenol	ug/L	200	36	98	35	10	135	115
Pyrene	ug/L	200	39	108	38	37	132	45
1,2,4-Trichlorobenzene	ug/L	200	39	97	32	10	142	52
Soil			**LCS**			**Spike**		
Compound	**Units**	**Spike Level**	**LCL**	**UCL**	**RPD**	**LCL**	**UCL**	**RPD**
Acenaphthene	ug/kg	6667	38	112	51	15	130	50
4-Bromophenyl phenyl ether	ug/kg	6667	46	120	81	27	136	48
Butyl benzyl phthalate	ug/kg	6667	47	115	54	27	130	48
4-Chloro-3-methylphenol	ug/kg	6667	39	111	52	16	128	52
2-Chlorophenol	ug/kg	6667	38	109	62	16	120	54
1,4-Dichlorobenzene	ug/kg	6667	36	107	57	20	105	62
2,4-Dinitrotoluene	ug/kg	6667	35	117	50	15	132	49
Hexachloroethane	ug/kg	6667	40	106	53	13	111	63
4-Methylphenol	ug/kg	13334	41	117	87	17	131	50
Naphthalene	ug/kg	6667	44	109	64	10	140	56
4-Nitrophenol	ug/kg	6667	30	125	43	10	154	88
N-Nitrosodi-n-propylamine	ug/kg	6667	36	114	45	30	118	51
Pentachlorophenol	ug/kg	6667	21	127	52	10	136	123
Phenol	ug/kg	6667	36	110	55	19	119	50
Pyrene	ug/kg	6667	43	118	48	10	168	69
1,2,4-Trichlorobenzene	ug/kg	6667	37	111	58	21	118	49

All samples are spiked with full analytes and the above compounds are the control analytes.
Samples extracted for QL method are prepared at the time of analysis at a 10X dilution.

Table 10 TCLP LCS Compounds	
LCS Compounds	Spiking Level, ng/µL in extract[1]
1,4-Dichlorobenzene	100
2,4-Dinitrotoluene	100
Hexachlorobenzene	100
Hexachlorobutadiene	100
Hexachloroethane	100
2-Methylphenol	100
3 & 4-Methylphenol	200
Nitrobenzene	100
Pentachlorophenol	100
Pyridine	100
2,4,5-Trichlorophenol	100
2,4,6-Trichlorophenol	100

[1] Levels are 50 ng/µL if 2 µL injection is used

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department.

Table 11 8270C Surrogate Compounds			
Surrogate Compounds	Routine 8270C Spiking Concentration, ug/mL	Low Level 8270C Spiked Method Code 42	Low Level 8270C Spiked Method Code QL
Nitrobenzene-d5	100	20	200
2-Fluorobiphenyl	100	20	200
Terphenyl-d14	100	20	200
1,2-Dichlorobenzene-d4[1]	100	20	200
Phenol-d6	150	30	300
2-Fluorophenol	150	30	300
2,4,6-Tribromophenol	150	30	300
2-Chlorophenol-d4[1]	150	30	300

[1] Included in standard mix, but not routinely evaluated for method 8270C

Samples extracted for QL method are prepared at the time of analysis at a 10X dilution.

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12

Calibration Levels, Primary Standard, ug/ml (for 2ul injection)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Pyridine	10	20	25	40	60	80
N-nitrosodimethylamine	10	20	25	40	60	80
Aniline	10	20	25	40	60	80
Phenol	10	20	25	40	60	80
Bis(2-chloroethyl)ether	10	20	25	40	60	80
2-Chlorophenol	10	20	25	40	60	80
1,3-Dichlorobenzene	10	20	25	40	60	80
1,4-Dichlorobenzene	10	20	25	40	60	80
Benzyl alcohol	10	20	25	40	60	80
1,2-Dichlorobenzene	10	20	25	40	60	80
2-Methylphenol	10	20	25	40	60	80
2,2'-oxybis(1-chloropropane)[1]	10	20	25	40	60	80
4-Methylphenol	10	20	25	40	60	80
N-Nitroso-di-n-propylamine	10	20	25	40	60	80
Hexachloroethane	10	20	25	40	60	80
Nitrobenzene	10	20	25	40	60	80
Isophorone	10	20	25	40	60	80
2-Nitrophenol	10	20	25	40	60	80
2,4-Dimethylphenol	10	20	25	40	60	80
Benzoic acid	10	20	25	40	60	80
Bis(2-chloroethoxy)methane	10	20	25	40	60	80
2,4-Dichlorophenol	10	20	25	40	60	80
1,2,4-Trichlorobenzene	10	20	25	40	60	80
Naphthalene	10	20	25	40	60	80
4-Chloroaniline	10	20	25	40	60	80
Hexachlorobutadiene	10	20	25	40	60	80
4-Chloro-3-methylphenol	10	20	25	40	60	80
2-Methylnaphthalene	10	20	25	40	60	80
Hexachlorocyclopentadiene	10	20	25	40	60	80
2,4,6-Trichlorophenol	10	20	25	40	60	80
2,4,5-Trichlorophenol	10	20	25	40	60	80
2-Chloronaphthalene	10	20	25	40	60	80
2-Nitroaniline	10	20	25	40	60	80
Dimethyl phthalate	10	20	25	40	60	80
Acenaphthylene	10	20	25	40	60	80
3-Nitroaniline	10	20	25	40	60	80
Acenaphthene	10	20	25	40	60	80
2,4-Dinitrophenol	10	20	25	40	60	80
4-Nitrophenol	10	20	25	40	60	80
Dibenzofuran	10	20	25	40	60	80

Table 12

Calibration Levels, Primary Standard, ug/ml (for 2ul injection)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
2,4-Dinitrotoluene	10	20	25	40	60	80
2,6-Dinitrotoluene	10	20	25	40	60	80
Diethylphthalate	10	20	25	40	60	80
4-Chlorophenyl phenyl ether	10	20	25	40	60	80
Fluorene	10	20	25	40	60	80
4-Nitroaniline	10	20	25	40	60	80
4,6-Dinitro-2-methylphenol	10	20	25	40	60	80
N-Nitrosodiphenylamine	10	20	25	40	60	80
Azobenzene[2]	10	20	25	40	60	80
4-Bromophenyl phenyl ether	10	20	25	40	60	80
Hexachlorobenzene	10	20	25	40	60	80
Pentachlorophenol	10	20	25	40	60	80
Phenanthrene	10	20	25	40	60	80
Anthracene	10	20	25	40	60	80
Carbazole	10	20	25	40	60	80
Di-n-butyl phthalate	10	20	25	40	60	80
Fluoranthene	10	20	25	40	60	80
Benzidine	10	20	25	40	60	80
Pyrene	10	20	25	40	60	80
Butyl benzyl phthalate	10	20	25	40	60	80
3,3'-Dichlorobenzidine	10	20	25	40	60	80
Benzo(a)anthracene	10	20	25	40	60	80
Bis(2-ethylhexyl)phthalate	10	20	25	40	60	80
Chrysene	10	20	25	40	60	80
Di-n-octylphthalate	10	20	25	40	60	80
Benzo(b)fluoranthene	10	20	25	40	60	80
Benzo(k)fluoranthene	10	20	25	40	60	80
Benzo(a)pyrene	10	20	25	40	60	80
Indeno(1,2,3-cd)pyrene	10	20	25	40	60	80
Dibenz(a,h)anthracene	10	20	25	40	60	80
Benzo(g,h,i)perylene	10	20	25	40	60	80

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2]Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 12A - Calibration Levels for Low Level, ug/ml (for 2ul injection)

Analytes	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Pyridine	0.2	1.0	2.0	5.0	10.0	20.0	40
N-nitrosodimethylamine	0.2	1.0	2.0	5.0	10.0	20.0	40
Aniline	0.2	1.0	2.0	5.0	10.0	20.0	40
Phenol	0.2	1.0	2.0	5.0	10.0	20.0	40
Bis(2-chloroethyl)ether	0.2	1.0	2.0	5.0	10.0	20.0	40
2-Chlorophenol	0.2	1.0	2.0	5.0	10.0	20.0	40
1,3-Dichlorobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
1,4-Dichlorobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
Benzyl alcohol	0.2	1.0	2.0	5.0	10.0	20.0	40
1,2-Dichlorobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
2-Methylphenol	0.2	1.0	2.0	5.0	10.0	20.0	40
2,2'-oxybis(1-chloropropane)[1]	0.2	1.0	2.0	5.0	10.0	20.0	40
4-Methylphenol	0.2	1.0	2.0	5.0	10.0	20.0	40
N-Nitroso-di-n-propylamine	0.2	1.0	2.0	5.0	10.0	20.0	40
Hexachloroethane	0.2	1.0	2.0	5.0	10.0	20.0	40
Nitrobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
Isophorone	0.2	1.0	2.0	5.0	10.0	20.0	40
2-Nitrophenol	0.2	1.0	2.0	5.0	10.0	20.0	40
2,4-Dimethylphenol	0.2	1.0	2.0	5.0	10.0	20.0	40
Benzoic acid	0.2	1.0	2.0	5.0	10.0	20.0	40
Bis(2-chloroethoxy)methane	0.2	1.0	2.0	5.0	10.0	20.0	40
2,4-Dichlorophenol	0.2	1.0	2.0	5.0	10.0	20.0	40
1,2,4-Trichlorobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
Naphthalene	0.2	1.0	2.0	5.0	10.0	20.0	40
4-Chloroaniline	0.2	1.0	2.0	5.0	10.0	20.0	40
Hexachlorobutadiene	0.2	1.0	2.0	5.0	10.0	20.0	40
4-Chloro-3-methylphenol	0.2	1.0	2.0	5.0	10.0	20.0	40
2-Methylnaphthalene	0.2	1.0	2.0	5.0	10.0	20.0	40
Hexachlorocyclopentadiene	0.2	1.0	2.0	5.0	10.0	20.0	40
2,4,6-Trichlorophenol	0.2	1.0	2.0	5.0	10.0	20.0	40
2,4,5-Trichlorophenol	0.2	1.0	2.0	5.0	10.0	20.0	40
2-Chloronaphthalene	0.2	1.0	2.0	5.0	10.0	20.0	40
2-Nitroaniline	0.2	1.0	2.0	5.0	10.0	20.0	40
Dimethyl phthalate	0.2	1.0	2.0	5.0	10.0	20.0	40
Acenaphthylene	0.2	1.0	2.0	5.0	10.0	20.0	40
3-Nitroaniline	0.2	1.0	2.0	5.0	10.0	20.0	40
Acenaphthene	0.2	1.0	2.0	5.0	10.0	20.0	40
2,4-Dinitrophenol	1.0	2.0	5.0	10.0	15.0	20.0	40
4-Nitrophenol	1.0	2.0	5.0	10.0	15.0	20.0	40
Dibenzofuran	0.2	1.0	2.0	5.0	10.0	20.0	40

Table 12A - Calibration Levels for Low Level, ug/ml (for 2ul injection)

Analytes	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2,4-Dinitrotoluene	0.2	1.0	2.0	5.0	10.0	20.0	40
2,6-Dinitrotoluene	0.2	1.0	2.0	5.0	10.0	20.0	40
Diethylphthalate	0.2	1.0	2.0	5.0	10.0	20.0	40
4-Chlorophenyl phenyl ether	0.2	1.0	2.0	5.0	10.0	20.0	40
Fluorene	0.2	1.0	2.0	5.0	10.0	20.0	40
4-Nitroaniline	0.2	1.0	2.0	5.0	10.0	20.0	40
4,6-Dinitro-2-methylphenol	0.2	1.0	2.0	5.0	10.0	20.0	40
N-Nitrosodiphenylamine	0.2	1.0	2.0	5.0	10.0	20.0	40
Azobenzene[2]	0.2	1.0	2.0	5.0	10.0	20.0	40
4-Bromophenyl phenyl ether	0.2	1.0	2.0	5.0	10.0	20.0	40
Hexachlorobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
Pentachlorophenol	1.0	2.0	5.0	10.0	15.0	20.0	40
Phenanthrene	0.2	1.0	2.0	5.0	10.0	20.0	40
Anthracene	0.2	1.0	2.0	5.0	10.0	20.0	40
Carbazole	0.2	1.0	2.0	5.0	10.0	20.0	40
Di-n-butyl phthalate	0.2	1.0	2.0	5.0	10.0	20.0	40
Fluoranthene	0.2	1.0	2.0	5.0	10.0	20.0	40
Benzidine	0.2	1.0	2.0	5.0	10.0	20.0	40
Pyrene	0.2	1.0	2.0	5.0	10.0	20.0	40
Butyl benzyl phthalate	0.2	1.0	2.0	5.0	10.0	20.0	40
3,3'-Dichlorobenzidine	0.2	1.0	2.0	5.0	10.0	20.0	40
Benzo(a)anthracene	0.2	1.0	2.0	5.0	10.0	20.0	40
Bis(2-ethylhexyl)phthalate	0.2	1.0	2.0	5.0	10.0	20.0	40
Chrysene	0.2	1.0	2.0	5.0	10.0	20.0	40
Di-n-octylphthalate	0.2	1.0	2.0	5.0	10.0	20.0	40
Benzo(b)fluoranthene	0.2	1.0	2.0	5.0	10.0	20.0	40
Benzo(k)fluoranthene	0.2	1.0	2.0	5.0	10.0	20.0	40
Benzo(a)pyrene	0.2	1.0	2.0	5.0	10.0	20.0	40
Indeno(1,2,3-cd)pyrene	0.2	1.0	2.0	5.0	10.0	20.0	40
Dibenz(a,h)anthracene	0.2	1.0	2.0	5.0	10.0	20.0	40
Benzo(g,h,i)perylene	0.2	1.0	2.0	5.0	10.0	20.0	40

Table 13 - Calibration Levels, Appendix IX Standard, µg/mL (for 2ul injection)

Analytes	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2-Picoline	0.2	1.0	2.0	5.0	10.0	20.0	40
N-Nitrosomethylethylamine	0.2	1.0	2.0	5.0	10.0	20.0	40
Methyl methanesulfonate	0.2	1.0	2.0	5.0	10.0	20.0	40
N-Nitrosodiethylamine	0.2	1.0	2.0	5.0	10.0	20.0	40
Ethyl methanesulfonate	0.2	1.0	2.0	5.0	10.0	20.0	40
Pentachloroethane	0.2	1.0	2.0	5.0	10.0	20.0	40
Acetophenone	0.2	1.0	2.0	5.0	10.0	20.0	40
N-Nitrosopyrrolidine	0.2	1.0	2.0	5.0	10.0	20.0	40
N-Nitrosomorpholine	0.2	1.0	2.0	5.0	10.0	20.0	40
o-Toluidine	0.2	1.0	2.0	5.0	10.0	20.0	40
3-Methylphenol	0.2	1.0	2.0	5.0	10.0	20.0	40
N-Nitrosopiperidine	0.2	1.0	2.0	5.0	10.0	20.0	40
o,o,o-Triethyl-Phosphorothioate	0.2	1.0	2.0	5.0	10.0	20.0	40
a,a-Dimethyl-phenethylamine	0.2	1.0	2.0	5.0	10.0	20.0	40
2,6-Dichlorophenol	0.2	1.0	2.0	5.0	10.0	20.0	40
Hexachloropropene	0.2	1.0	2.0	5.0	10.0	20.0	40
p-Phenylenediamine	0.2	1.0	2.0	5.0	10.0	20.0	40
n-Nitrosodi-n-butylamine	0.2	1.0	2.0	5.0	10.0	20.0	40
Safrole	0.2	1.0	2.0	5.0	10.0	20.0	40
1,2,4,5-Tetrachlorobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
Isosafrole 1 + 2	0.2	1.0	2.0	5.0	10.0	20.0	40
1,4-Dinitrobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
1,4-Naphthoquinone	0.2	1.0	2.0	5.0	10.0	20.0	40
1,3-Dinitrobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
Pentachlorobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
1-Naphthylamine	0.2	1.0	2.0	5.0	10.0	20.0	40
2-Naphthylamine	0.2	1.0	2.0	5.0	10.0	20.0	40
2,3,4,6-Tetrachlorophenol	0.2	1.0	2.0	5.0	10.0	20.0	40
5-Nitro-o-toluidine	0.2	1.0	2.0	5.0	10.0	20.0	40
Thionazin	0.2	1.0	2.0	5.0	10.0	20.0	40
1,3,5-Trinitrobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
Sulfotepp	0.2	1.0	2.0	5.0	10.0	20.0	40
Phorate	0.2	1.0	2.0	5.0	10.0	20.0	40
Phenacetin	0.2	1.0	2.0	5.0	10.0	20.0	40
Diallate 1 + 2	0.2	1.0	2.0	5.0	10.0	20.0	40
Dimethoate	0.2	1.0	2.0	5.0	10.0	20.0	40
4-Aminobiphenyl	0.2	1.0	2.0	5.0	10.0	20.0	40
Pentachloronitrobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40

Table 13 - Calibration Levels, Appendix IX Standard, µg/mL (for 2ul injection)

Analytes	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Pronamide	0.2	1.0	2.0	5.0	10.0	20.0	40
Disulfoton	0.2	1.0	2.0	5.0	10.0	20.0	40
2-secbutyl-4,6-dinitrophenol (Dinoseb)	0.2	1.0	2.0	5.0	10.0	20.0	40
Methyl parathion	0.2	1.0	2.0	5.0	10.0	20.0	40
4-Nitroquinoline-1-oxide	0.2	1.0	2.0	5.0	10.0	20.0	40
Parathion	0.2	1.0	2.0	5.0	10.0	20.0	40
Isodrin	0.2	1.0	2.0	5.0	10.0	20.0	40
Kepone	0.2	1.0	2.0	5.0	10.0	20.0	40
Famphur	0.2	1.0	2.0	5.0	10.0	20.0	40
Methapyrilene	0.2	1.0	2.0	5.0	10.0	20.0	40
Aramite 1 and 2	0.2	1.0	2.0	5.0	10.0	20.0	40
p-(Dimethylamino)azobenzene	0.2	1.0	2.0	5.0	10.0	20.0	40
p-Chlorobenzilate	0.2	1.0	2.0	5.0	10.0	20.0	40
3,3'-Dimethylbenzidine	0.2	1.0	2.0	5.0	10.0	20.0	40
2-Acetylaminofluorene	0.2	1.0	2.0	5.0	10.0	20.0	40
Dibenz (a,j)acridine	0.2	1.0	2.0	5.0	10.0	20.0	40
7,12-Dimethylbenz(a)anthracene	0.2	1.0	2.0	5.0	10.0	20.0	40
3-Methylcholanthrene	0.2	1.0	2.0	5.0	10.0	20.0	40

Table 14
Method 8270C Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Acenaphthene	50	27.6	60.1-132.3
Acenaphthylene	50	40.2	53.5-126.0
Aldrin[1]	50	39.0	7.2-152.2
Anthracene	50	32.0	43.4-118.0
Benz(a)anthracene	50	27.6	41.8-133.0
Benzo(b)fluoranthene	50	38.8	42.0-140.4
Benzo(k)fluoranthene	50	32.3	25.2-145.7
Benzo(a)pyrene	50	39.0	31.7-148.0
Benzo(ghi)perylene	50	58.9	D-195.0
Benzylbutyl phthalate	50	23.4	D-139.9
B-BHC[1]	50	31.5	41.5-130.6
d-BHC[1]	50	21.6	D-100.0
Bis(2-chloroethyl) ether	50	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	50	34.5	49.2-164.7

Table 14
Method 8270C Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Bis(2-chloroisopropyl) ether	50	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	50	41.1	28.9-136.8
4-Bromophenyl phenyl ether	50	23.0	64.9-114.4
2-Chloronaphthalene	50	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	50	33.4	38.4-144.7
Chrysene	50	48.3	44.1-139.9
4,4'-DDD[1]	50	31.0	D-134.5
4,4'-DDE[1]	50	32.0	19.2-119.7
4,4'-DDT[1]	50	61.6	D-170.6
Dibenzo(a,h)anthracene	50	70.0	D-199.7
Di-n-butyl phthalate	50	16.7	8.4-111.0
1,2-Dichlorobenzene	50	30.9	48.6-112.0
1,3-Dichlorobenzene	50	41.7	16.7-153.9
1,4-Dichlorobenzene	50	32.1	37.3-105.7
3,3'-Dichlorobenzidine	50	71.4	8.2-212.5
Dieldrin[1]	50	30.7	44.3-119.3
Diethyl phthalate	50	26.5	D-100.0
Dimethyl phthalate	50	23.2	D-100.0
2,4-Dinitrotoluene	50	21.8	47.5-126.9
2,6-Dinitrotoluene	50	29.6	68.1-136.7
Di-n-octylphthalate	50	31.4	18.6-131.8
Endosulfan sulfate[1]	50	16.7	D-103.5
Endrin aldehyde	50	32.5	D-188.8
Fluoranthene	50	32.8	42.9-121.3
Fluorene	50	20.7	71.6-108.4
Heptachlor[1]	50	37.2	D-172.2
Heptachlor epoxide[1]	50	54.7	70.9-109.4
Hexachlorobenzene	50	24.9	7.8-141.5
Hexachlorobutadiene	50	26.3	37.8-102.2
Hexachloroethane	50	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	50	44.6	D-150.9
Isophorone	50	63.3	46.6-180.2
Naphthalene	50	30.1	35.6-119.6
Nitrobenzene	50	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	50	55.4	13.6-197.9
PCB-1260[1]	50	54.2	19.3-121.0
Phenanthrene	50	20.6	65.2-108.7
Pyrene	50	25.2	69.6-100.0

Table 14
Method 8270C Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
1,2,4-Trichlorobenzene	50	28.1	57.3-129.2
4-Chloro-3-methylphenol	50	37.2	40.8-127.9
2-Chlorophenol	50	28.7	36.2-120.4
2,4-Chlorophenol	50	26.4	52.5-121.7
2,4-Dimethylphenol	50	26.1	41.8-109.0
2,4-Dinitrophenol	50	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	50	93.2	53.0-100.0
2-Nitrophenol	50	35.2	45.0-166.7
4-Nitrophenol	50	47.2	13.0-106.5
Pentachlorophenol	50	48.9	38.1-151.8
Phenol	50	22.6	16.6-100.0
2,4,6-Trichlorophenol	50	31.7	52.4-129.2

[1]Since the organochlorine pesticides and PCBs are normally determined by method 8081A at TestAmerica, Pittsburgh they will not be included in the initial demonstration of capability for method 8270C.

Table 14A
Method 8270C Low Level Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Acenaphthene	20	27.6	60.1-132.3
Acenaphthylene	20	40.2	53.5-126.0
Aldrin[1]	20	39.0	7.2-152.2
Anthracene	20	32.0	43.4-118.0
Benz(a)anthracene	20	27.6	41.8-133.0
Benzo(b)fluoranthene	20	38.8	42.0-140.4
Benzo(k)fluoranthene	20	32.3	25.2-145.7
Benzo(a)pyrene	20	39.0	31.7-148.0
Benzo(ghi)perylene	20	58.9	D-195.0
Benzylbutyl phthalate	20	23.4	D-139.9
B-BHC[1]	20	31.5	41.5-130.6
d-BHC[1]	20	21.6	D-100.0
Bis(2-chloroethyl) ether	20	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	20	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	20	46.3	62.8-138.6

Table 14A
Method 8270C Low Level Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Bis(2-ethylhexyl) phthalate	20	41.1	28.9-136.8
4-Bromophenyl phenyl ether	20	23.0	64.9-114.4
2-Chloronaphthalene	20	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	20	33.4	38.4-144.7
Chrysene	20	48.3	44.1-139.9
4,4'-DDD[1]	20	31.0	D-134.5
4,4'-DDE[1]	20	32.0	19.2-119.7
4,4'-DDT[1]	20	61.6	D-170.6
Dibenzo(a,h)anthracene	20	70.0	D-199.7
Di-n-butyl phthalate	20	16.7	8.4-111.0
1,2-Dichlorobenzene	20	30.9	48.6-112.0
1,3-Dichlorobenzene	20	41.7	16.7-153.9
1,4-Dichlorobenzene	20	32.1	37.3-105.7
3,3'-Dichlorobenzidine	20	71.4	8.2-212.5
Dieldrin[1]	20	30.7	44.3-119.3
Diethyl phthalate	20	26.5	D-100.0
Dimethyl phthalate	20	23.2	D-100.0
2,4-Dinitrotoluene	20	21.8	47.5-126.9
2,6-Dinitrotoluene	20	29.6	68.1-136.7
Di-n-octylphthalate	20	31.4	18.6-131.8
Endosulfan sulfate[1]	20	16.7	D-103.5
Endrin aldehyde	20	32.5	D-188.8
Fluoranthene	20	32.8	42.9-121.3
Fluorene	20	20.7	71.6-108.4
Heptachlor[1]	20	37.2	D-172.2
Heptachlor epoxide[1]	20	54.7	70.9-109.4
Hexachlorobenzene	20	24.9	7.8-141.5
Hexachlorobutadiene	20	26.3	37.8-102.2
Hexachloroethane	20	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	20	44.6	D-150.9
Isophorone	20	63.3	46.6-180.2
Naphthalene	20	30.1	35.6-119.6
Nitrobenzene	20	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	20	55.4	13.6-197.9
PCB-1260[1]	20	54.2	19.3-121.0
Phenanthrene	20	20.6	65.2-108.7
Pyrene	20	25.2	69.6-100.0
1,2,4-Trichlorobenzene	20	28.1	57.3-129.2

Table 14A
Method 8270C Low Level Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
4-Chloro-3-methylphenol	20	37.2	40.8-127.9
2-Chlorophenol	20	28.7	36.2-120.4
2,4-Chlorophenol	20	26.4	52.5-121.7
2,4-Dimethylphenol	20	26.1	41.8-109.0
2,4-Dinitrophenol	20	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	20	93.2	53.0-100.0
2-Nitrophenol	20	35.2	45.0-166.7
4-Nitrophenol	20	47.2	13.0-106.5
Pentachlorophenol	20	48.9	38.1-151.8
Phenol	20	22.6	16.6-100.0
2,4,6-Trichlorophenol	20	31.7	52.4-129.2

Table 15
Method 8270C Surrogate QC Acceptance Criteria

	Water			Soil		
Compound	**AMT ug/L**	**LCL**	**UCL**	**AMT ug/kg**	**LCL**	**UCL**
2-Fluorobiphenyl	50	35	115	1665	26	128
2-Fluorophenol	75	10	118	2498	34	115
2,4,6-Tribromophenol	75	19	138	2498	21	144
Nitrobenzene-d5	50	39	115	1665	30	118
Phenol-d5	75	18	115	2498	35	117
Terphenyl-d14	50	17	129	1665	40	115

The acceptance criteria listed above is based on laboratory generated data.

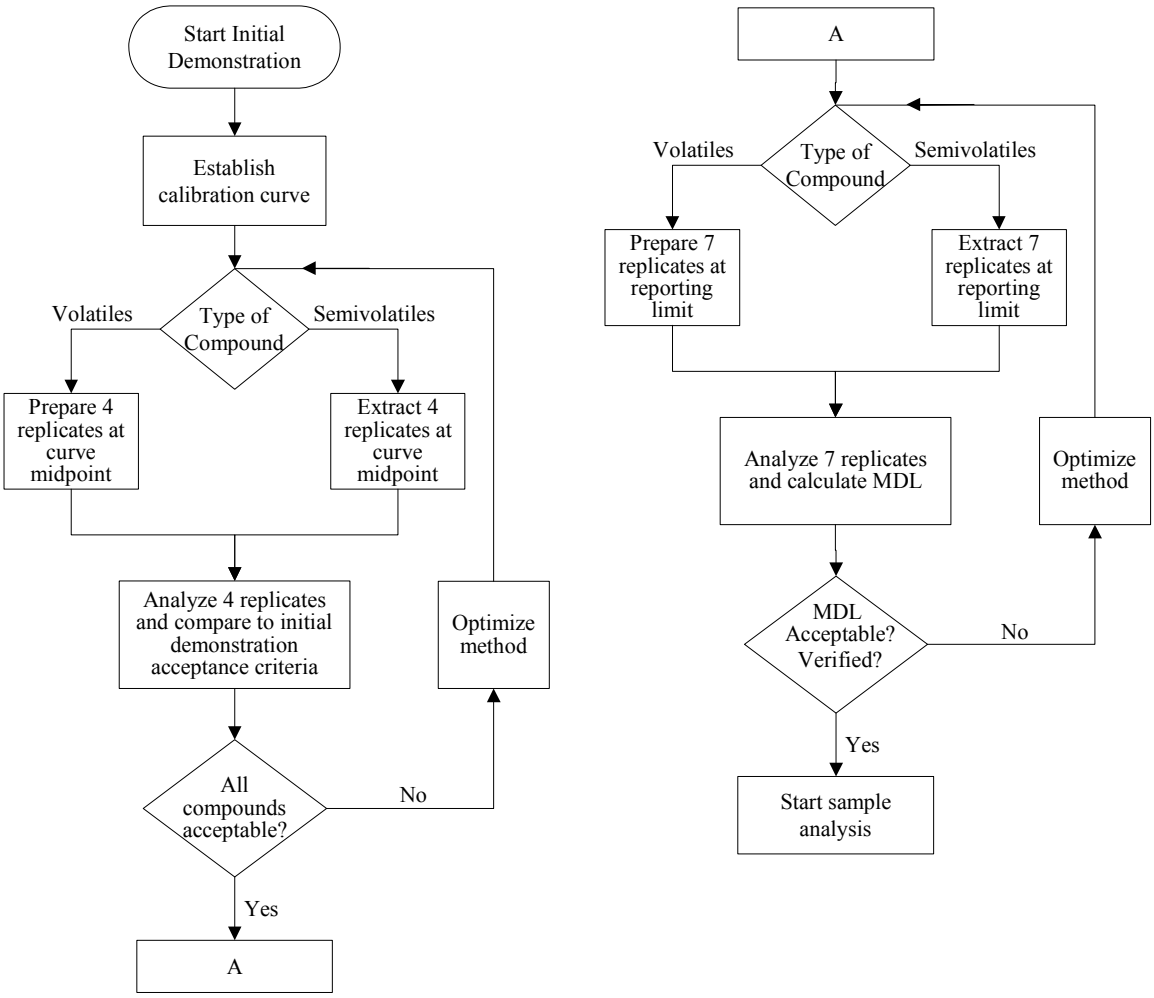
Table 15A
Method 8270C Low Level (Method Code 42) Surrogate QC Acceptance Criteria

	Water			Soil		
Compound	**AMT ug/L**	**LCL**	**UCL**	**AMT ug/kg**	**LCL**	**UCL**
2-Fluorobiphenyl	20	19	107	667	28	108
2-Fluorophenol	30	10	111	1000	28	107
2,4,6-Tribromophenol	30	16	122	1000	21	116
Nitrobenzene-d5	20	23	112	667	27	110
Phenol-d5	30	15	112	1000	30	112
Terphenyl-d14	20	10	132	667	21	130

Table 15B Method 8270C Low Level (Method Code QL) Surrogate QC Acceptance Criteria						
	Water			Soil		
Compound	AMT ug/L	LCL	UCL	AMT ug/kg	LCL	UCL
2-Fluorobiphenyl	200	27	104	6667	20	109
2-Fluorophenol	300	17	102	10000	10	113
2,4,6-Tribromophenol	300	20	107	10000	10	117
Nitrobenzene-d5	200	33	103	6667	18	106
Phenol-d5	300	25	107	10000	18	113
Terphenyl-d14	200	14	127	6667	10	138

Samples extracted for QL method are prepared at the time of analysis at a 10X dilution.

17.2 Initial demonstration and MDL[i]



[i] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

ATTACHMENT A

MODIFICATIONS REQUIRED FOR ANALYSIS OF WASTEWATER FOLLOWING METHOD 625

19. REQUIREMENTS FOR METHOD 625

19.1. Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits. The standard analyte list and reporting limits are listed in Table A-1.

19.2. This method can be applied only to aqueous matrices.

19.3. EPA has approved modification to method 625: one extraction can be done. In using single pH extractions for 625 the laboratory should analyze a series of LCSs and have the recovery and precision data filed and readily available. Refer to Appendix A.

19.4. The tune period for this method is defined as 24 hours.

19.5. Initial calibration curve requirements:

19.5.1. The initial calibration curve for this method requires at least three points.

19.5.2. Target compounds must have RSD ≤ 35%.

19.5.3. If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds.

19.6. Continuing calibration verification requirements: All target compounds must have %D ≤ 20%.

19.7. Matrix Spike and LCS requirements:

19.7.1. A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

19.8. The laboratory must, on an ongoing basis, spike at least 5% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to 20 samples per month, at least one spiked sample per month is required. The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control.

19.8.1. If any parameter fails the acceptance criteria for recovery, a QC check standard

containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of single-component parameters in must be measured in the sample, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spike sample.

Table A-1

TestAmerica Pittsburgh Method 625 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	50
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10

Table A-1

TestAmerica Pittsburgh Method 625 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous µg/L
Pentachlorophenol	87-86-5	50
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10

Table A-2
Method 625 LCS and MS Compounds and Spike Concentrations

LCS Compounds	Spiking Level, ng in injected 2 µL injection
Phenol	100
Bis(2-chloroethyl)ether	100
2-Chlorophenol	100
1,3-Dichlorobenzene	100
1,4-Dichlorobenzene	100
1,2-Dichlorobenzene	100
2,2'-oxybis(1-chloropropane)	100
N-Nitroso-di-n-propylamine	100
Hexachloroethane	100
Nitrobenzene	100
Isophorone	100
2-Nitrophenol	100
2,4-Dimethylphenol	100
Bis(2-chloroethoxy)methane	100
2,4-Dichlorophenol	100
1,2,4-Trichlorobenzene	100
Naphthalene	100
Hexachlorobutadiene	100
4-Chloro-3-methylphenol	100
Hexachlorocyclopentadiene	100
2,4,6-Trichlorophenol	100
2-Chloronaphthalene	100
Dimethyl phthalate	100
Acenaphthylene	100
Acenaphthene	100
2,4-Dinitrophenol	100
4-Nitrophenol	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Diethylphthalate	100
4-Chlorophenyl phenyl ether	100
Fluorene	100
4,6-Dinitro-2-methylphenol	100
N-Nitrosodiphenylamine	100
4-Bromophenyl phenyl ether	100
Hexachlorobenzene	100
Pentachlorophenol	100

Table A-2 Method 625 LCS and MS Compounds and Spike Concentrations	
LCS Compounds	Spiking Level, ng in injected 2 µL injection
Phenanthrene	100
Anthracene	100
Di-n-butyl phthalate	100
Fluoranthene	100
Benzidine	100
Pyrene	100
Butyl benzyl phthalate	100
3,3'-Dichlorobenzidine	100
Benzo(a)anthracene	100
Bis(2-ethylhexyl)phthalate	100
Chrysene	100
Di-n-octylphthalate	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Indeno(1,2,3-cd)pyrene	100
Dibenz(a,h)anthracene	100
Benzo(g,h,i)perylene	100

TABLE A-3 Method 625 LCS and MS Compounds and Spike Concentrations	
Surrogate Compounds	Spiking Level, ug/L in extract
Nitrobenzene-d5	50
2-Fluorobiphenyl	50
Terphenyl-d14	50
2-Fluorophenol	50
2,4,6-Tribromophenol	50
Phenol-d_5	50

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

TABLE A-4. METHOD 625 LCS, MS AND SURROGATE QC ACCEPTANCE CRITERIA

Compound	MDL[1] (ug/L)	LCS			Matrix Spike		
		LCL	UCL	RPD	LCL	UCL	RPD
1,2,4-Trichlorobenzene	1.377	31	110	37	31	110	37
1,2-Dichlorobenzene	1.361	32	129	20	32	129	20
1,2-Diphenylhydrazine	1.339	30	125	25	30	125	25
1,3-Dichlorobenzene	1.269	1	172	35	1	172	35
1,4-Dichlorobenzene	1.317	28	110	36	28	110	36
2,2'-oxybis(1-Chloropropane)	1.715	50	150	50	50	150	50
2,4,6-Trichlorophenol	1.497	46	135	27	46	135	27
2,4-Dichlorophenol	1.335	42	115	44	42	115	44
2,4-Dimethylphenol	1.833	32	119	20	32	119	20
2,4-Dinitrophenol	14.763	1	191	53	1	191	53
2,4-Dinitrotoluene	1.289	47	131	32	47	131	32
2,6-Dinitrotoluene	1.4	50	158	20	50	158	20
2-Chloronaphthalene	1.429	60	118	20	60	118	20
2-Chlorophenol	1.389	19	124	43	19	124	43
2-Methyl-4,6-dinitrophenol	9.644	10	181	40	10	181	40
2-Nitrophenol	2.99	29	182	32	29	182	32
3,3'-Dichlorobenzidine	25.023	1	162	56	1	162	56
4-Bromophenyl phenyl ether	1.238	53	127	20	53	127	20
4-Chloro-3-methylphenol	1.314	29	124	55	29	124	55
4-Chlorophenyl phenyl ether	1.629	25	158	27	25	158	27
4-Nitrophenol	1.775	19	144	34	19	144	34
Acenaphthene	1.556	39	118	35	39	118	35
Acenaphthylene	1.822	33	145	23	33	145	23
Anthracene	1.195	27	133	22	27	133	22
Benzidine	1.998	1	140	50	1	140	50
Benzo(a)anthracene	0.932	33	143	23	33	143	23

TABLE A-4. METHOD 625 LCS, MS AND SURROGATE QC ACCEPTANCE CRITERIA

Compound	MDL[1] (ug/L)	LCS			Matrix Spike		
		LCL	UCL	RPD	LCL	UCL	RPD
Benzo(a)pyrene	2.503	17	163	31	17	163	31
Benzo(b)fluoranthene	0.857	24	159	28	24	159	28
Benzo(ghi)perylene	0.997	1	219	50	1	219	50
Benzo(k)fluoranthene	1.101	11	162	31	11	162	31
bis(2-Chloroethoxy)methane	3.45	33	184	30	33	184	30
bis(2-Chloroethyl) ether	1.44	12	158	30	12	158	30
bis(2-Ethylhexyl) phthalate	0.907	8	158	31	8	158	31
Butyl benzyl phthalate	1.011	1	152	35	1	152	35
Chrysene	0.953	17	168	31	17	168	31
Dibenzo(a,h)anthracene	1.039	1	227	55	1	227	55
Diethyl phthalate	1.13	1	114	24	1	114	24
Dimethyl phthalate	1.251	1	112	22	1	112	22
Di-n-butyl phthalate	1.104	1	118	24	1	118	24
Di-n-octyl phthalate	0.948	4	146	29	4	146	29
Fluoranthene	1.124	26	137	23	26	137	23
Fluorene	1.548	59	121	20	59	121	20
Hexachlorobenzene	1.261	57	128	22	57	128	22
Hexachlorobutadiene	1.47	36	116	32	36	116	32
Hexachlorocyclopentadiene	6.259	1	138	54	1	138	54
Hexachloroethane	1.371	30	110	33	30	110	33
Indeno(1,2,3-cd)pyrene	0.998	1	171	37	1	171	37
Isophorone	1.404	21	196	37	21	196	37
Naphthalene	1.5	21	133	23	21	133	23
Nitrobenzene	1.455	45	130	50	45	130	50
N-Nitrosodimethylamine	1.694	1	230	47	1	230	47
N-Nitrosodi-n-propylamine	1.538	30	115	36	30	115	36

TABLE A-4. METHOD 625 LCS, MS AND SURROGATE QC ACCEPTANCE CRITERIA

Compound	MDL[1] (ug/L)	LCS			Matrix Spike		
		LCL	UCL	RPD	LCL	UCL	RPD
N-Nitrosodiphenylamine	4.191	5	138	68	5	138	68
Pentachlorophenol	0.816	10	140	56	10	140	56
Phenanthrene	1.068	54	120	20	54	120	20
Phenol	1.98	10	131	43	10	131	43
Pyrene	0.941	46	130	31	46	130	31
Surrogates:							
2-Fluorobiphenyl		30	110		30	110	
2-Fluorophenol		13	110		13	110	
2,4,6-Tribromophenol		21	122		21	122	
Nitrobenzene-d5		32	112		32	112	
Phenol-d5		10	113		10	113	
Terphenyl-d14		10	144		10	144	

Note: The control limits are derived from laboratory generated data.

[1] The MDL listed are subject to change.

Attachment B
Standard Preparation

STL Pittsburgh

Standards Preparation Logbook Record

Nov-22-2005

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0806-05, STD20(10ug/ml) 8270/clp/625 — Analyst: bachas

Solvent: Methylene chloride Lot No.: b32e12 — Volume (ml): 1.0000
Date Prep./Opened: 10-27-2005
Date Expires(1): 04-27-2006 (6 Months)
Date Expires(2): 11-30-2006 (None)

Parent Std No.: BNA0391-05, 1,4 Dioxane — Aliquot Amount (ml): 0.0050
Parent Date Expires(1): 05-20-2006 Parent Date Expires(2): 06-30-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1,4-dioxan	2,000.0	10.000

Parent Std No.: BNA0526-05, EPA 8270 Benzidines Mix — Aliquot Amount (ml): 0.0050
Parent Date Expires(1): 07-06-2006 Parent Date Expires(2): 02-28-2008

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
3,3'-Dichlorobenzidine	2,000.0	10.000
3,3'-dimethylbenzidine	2,000.0	10.000
Benzidine	2,000.0	10.000

Parent Std No.: BNA0773-05, 8270/CLP/625 Stock Mix 100ug/ml — Aliquot Amount (ml): 0.1000

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
N-Nitrosodiphenylamine	100.00	10.000
2,4,6-Tribromophenol	100.00	10.000
2-Chlorophenol-d4	100.00	10.000
2-Fluorophenol	100.00	10.000
Phenol-d5	100.00	10.000
1,2-Dichlorobenzene-d4	100.00	10.000
2-Fluorobiphenyl	100.00	10.000
Nitrobenzene-d5	100.00	10.000
p-Terphenyl-d14	100.00	10.000
Multi-Component	100.00	10.000
Aniline	100.00	10.000
Benzoic Acid	100.00	10.000
Benzyl Alcohol	100.00	10.000
Pyridine	100.00	10.000

Parent Std No.: BNA0812-05, CLP SOW OLM04 Semivolatiles — Aliquot Amount (ml): 0.0050
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 02-28-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Acetophenone	2,000.0	10.000
Atrazine	2,000.0	10.000
Benzaldehyde	2,000.0	10.000

Biphenyl	2,000.0	10.000
Caprolactam	2,000.0	10.000

Parent Std No.: BNA0813-05, Equity 8270 ADD-ON Mix Aliquot Amount (ml): 0.0050
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 11-30-2006

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1-Methylnaphthalene	2,000.0	10.000
2,3,4,6-Tetrachlorophenol	2,000.0	10.000
2,3,5,6-Tetrachlorophenol	2,000.0	10.000
2-Naphthylamine	2,000.0	10.000
7,12-Dimethylbenz(a)anthracene	2,000.0	10.000
Methyl methanesulfonate	2,000.0	10.000
N-Nitroso-di-n-butylamine	2,000.0	10.000

Reviewed By: _____

STL Pittsburgh

Standards Preparation Logbook Record

Nov-22-2005

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0807-05, STD40(20ug/ml) 8270/clp/625 — Analyst: bachas

Solvent: Methylene chloride Lot No.: b32e12 — Volume (ml): 1.0000
Date Prep./Opened: 10-27-2005
Date Expires(1): 04-27-2006 (6 Months)
Date Expires(2): 11-30-2006 (None)

Parent Std No.: BNA0391-05, 1,4 Dioxane — Aliquot Amount (ml): 0.0100
Parent Date Expires(1): 05-20-2006 Parent Date Expires(2): 06-30-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1,4-dioxan	2,000.0	20.000

Parent Std No.: BNA0526-05, EPA 8270 Benzidines Mix — Aliquot Amount (ml): 0.0100
Parent Date Expires(1): 07-06-2006 Parent Date Expires(2): 02-28-2008

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
3,3'-Dichlorobenzidine	2,000.0	20.000
3,3'-dimethylbenzidine	2,000.0	20.000
Benzidine	2,000.0	20.000

Parent Std No.: BNA0773-05, 8270/CLP/625 Stock Mix 100ug/ml — Aliquot Amount (ml): 0.2000

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
N-Nitrosodiphenylamine	100.00	20.000
2,4,6-Tribromophenol	100.00	20.000
2-Chlorophenol-d4	100.00	20.000
2-Fluorophenol	100.00	20.000
Phenol-d5	100.00	20.000
1,2-Dichlorobenzene-d4	100.00	20.000
2-Fluorobiphenyl	100.00	20.000
Nitrobenzene-d5	100.00	20.000
p-Terphenyl-d14	100.00	20.000
Multi-Component	100.00	20.000
Aniline	100.00	20.000
Benzoic Acid	100.00	20.000
Benzyl Alcohol	100.00	20.000
Pyridine	100.00	20.000

Parent Std No.: BNA0812-05, CLP SOW OLM04 Semivolatiles — Aliquot Amount (ml): 0.0100
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 02-28-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Acetophenone	2,000.0	20.000
Atrazine	2,000.0	20.000
Benzaldehyde	2,000.0	20.000

Biphenyl	2,000.0	20.000
Caprolactam	2,000.0	20.000

Parent Std No.: BNA0813-05, Equity 8270 ADD-ON Mix Aliquot Amount (ml): 0.0100
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 11-30-2006

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1-Methylnaphthalene	2,000.0	20.000
2,3,4,6-Tetrachlorophenol	2,000.0	20.000
2,3,5,6-Tetrachlorophenol	2,000.0	20.000
2-Naphthylamine	2,000.0	20.000
7,12-Dimethylbenz(a)anthracene	2,000.0	20.000
Methyl methanesulfonate	2,000.0	20.000
N-Nitroso-di-n-butylamine	2,000.0	20.000

Reviewed By: _____

STL Pittsburgh

Standards Preparation Logbook Record

Nov-22-2005

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0805-05, STD50(25ug/ml) 8270/clp/625 — Analyst: bachas

Solvent: Methylene chloride Lot No.: b32e12 Volume (ml): 1.0000
Date Prep./Opened: 10-27-2005
Date Expires(1): 11-03-2005 (1 Week)
Date Expires(2): 11-30-2006 (None)

Parent Std No.: BNA0391-05, 1,4 Dioxane Aliquot Amount (ml): 0.0125
Parent Date Expires(1): 05-20-2006 Parent Date Expires(2): 06-30-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1,4-dioxan	2,000.0	25.000

Parent Std No.: BNA0526-05, EPA 8270 Benzidines Mix Aliquot Amount (ml): 0.0125
Parent Date Expires(1): 07-06-2006 Parent Date Expires(2): 02-28-2008

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
3,3'-Dichlorobenzidine	2,000.0	25.000
3,3'-dimethylbenzidine	2,000.0	25.000
Benzidine	2,000.0	25.000

Parent Std No.: BNA0773-05, 8270/CLP/625 Stock Mix 100ug/ml Aliquot Amount (ml): 0.2500

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
N-Nitrosodiphenylamine	100.00	25.000
2,4,6-Tribromophenol	100.00	25.000
2-Chlorophenol-d4	100.00	25.000
2-Fluorophenol	100.00	25.000
Phenol-d5	100.00	25.000
1,2-Dichlorobenzene-d4	100.00	25.000
2-Fluorobiphenyl	100.00	25.000
Nitrobenzene-d5	100.00	25.000
p-Terphenyl-d14	100.00	25.000
Multi-Component	100.00	25.000
Aniline	100.00	25.000
Benzoic Acid	100.00	25.000
Benzyl Alcohol	100.00	25.000
Pyridine	100.00	25.000

Parent Std No.: BNA0812-05, CLP SOW OLM04 Semivolatiles Aliquot Amount (ml): 0.0125
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 02-28-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Acetophenone	2,000.0	25.000
Atrazine	2,000.0	25.000
Benzaldehyde	2,000.0	25.000

Biphenyl	2,000.0	25.000
Caprolactam	2,000.0	25.000

Parent Std No.: BNA0813-05, Equity 8270 ADD-ON Mix Aliquot Amount (ml): 0.0125
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 11-30-2006

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1-Methylnaphthalene	2,000.0	25.000
2,3,4,6-Tetrachlorophenol	2,000.0	25.000
2,3,5,6-Tetrachlorophenol	2,000.0	25.000
2-Naphthylamine	2,000.0	25.000
7,12-Dimethylbenz(a)anthracene	2,000.0	25.000
Methyl methanesulfonate	2,000.0	25.000
N-Nitroso-di-n-butylamine	2,000.0	25.000

Reviewed By: _____

STL Pittsburgh

Standards Preparation Logbook Record

Nov-22-2005

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0808-05, STD80(40ug/ml) 8270/clp/625 Analyst: bachas

Solvent: Methylene chloride Lot No.: b32e12 Volume (ml): 1.0000
Date Prep./Opened: 10-27-2005
Date Expires(1): 04-27-2006 (6 Months)
Date Expires(2): 11-30-2006 (None)

Parent Std No.: BNA0391-05, 1,4 Dioxane Aliquot Amount (ml): 0.0200
Parent Date Expires(1): 05-20-2006 Parent Date Expires(2): 06-30-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1,4-dioxan	2,000.0	40.000

Parent Std No.: BNA0526-05, EPA 8270 Benzidines Mix Aliquot Amount (ml): 0.0200
Parent Date Expires(1): 07-06-2006 Parent Date Expires(2): 02-28-2008

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
3,3'-Dichlorobenzidine	2,000.0	40.000
3,3'-dimethylbenzidine	2,000.0	40.000
Benzidine	2,000.0	40.000

Parent Std No.: BNA0773-05, 8270/CLP/625 Stock Mix 100ug/ml Aliquot Amount (ml): 0.4000

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
N-Nitrosodiphenylamine	100.00	40.000
2,4,6-Tribromophenol	100.00	40.000
2-Chlorophenol-d4	100.00	40.000
2-Fluorophenol	100.00	40.000
Phenol-d5	100.00	40.000
1,2-Dichlorobenzene-d4	100.00	40.000
2-Fluorobiphenyl	100.00	40.000
Nitrobenzene-d5	100.00	40.000
p-Terphenyl-d14	100.00	40.000
Multi-Component	100.00	40.000
Aniline	100.00	40.000
Benzoic Acid	100.00	40.000
Benzyl Alcohol	100.00	40.000
Pyridine	100.00	40.000

Parent Std No.: BNA0812-05, CLP SOW OLM04 Semivolatiles Aliquot Amount (ml): 0.0200
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 02-28-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Acetophenone	2,000.0	40.000
Atrazine	2,000.0	40.000
Benzaldehyde	2,000.0	40.000

Page 1 of 2

Biphenyl	2,000.0	40.000
Caprolactam	2,000.0	40.000

Parent Std No.: BNA0813-05, Equity 8270 ADD-ON Mix — Aliquot Amount (ml): 0.0200
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 11-30-2006

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1-Methylnaphthalene	2,000.0	40.000
2,3,4,6-Tetrachlorophenol	2,000.0	40.000
2,3,5,6-Tetrachlorophenol	2,000.0	40.000
2-Naphthylamine	2,000.0	40.000
7,12-Dimethylbenz(a)anthracene	2,000.0	40.000
Methyl methanesulfonate	2,000.0	40.000
N-Nitroso-di-n-butylamine	2,000.0	40.000

Reviewed By: _____

STL Pittsburgh

Standards Preparation Logbook Record

Nov-22-2005

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0809-05, STD120(60ug/ml) 8270/clp/625 — Analyst: bachas

Solvent: Methylene chloride Lot No.: b32e12 — Volume (ml): 1.0000
Date Prep./Opened: 10-27-2005
Date Expires(1): 04-27-2006 (6 Months)
Date Expires(2): 11-30-2006 (None)

Parent Std No.: BNA0391-05, 1,4 Dioxane — Aliquot Amount (ml): 0.0300
Parent Date Expires(1): 05-20-2006 Parent Date Expires(2): 06-30-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1,4-dioxan	2,000.0	60.000

Parent Std No.: BNA0526-05, EPA 8270 Benzidines Mix — Aliquot Amount (ml): 0.0300
Parent Date Expires(1): 07-06-2006 Parent Date Expires(2): 02-28-2008

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
3,3'-Dichlorobenzidine	2,000.0	60.000
3,3'-dimethylbenzidine	2,000.0	60.000
Benzidine	2,000.0	60.000

Parent Std No.: BNA0773-05, 8270/CLP/625 Stock Mix 100ug/ml — Aliquot Amount (ml): 0.6000

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
N-Nitrosodiphenylamine	100.00	60.000
2,4,6-Tribromophenol	100.00	60.000
2-Chlorophenol-d4	100.00	60.000
2-Fluorophenol	100.00	60.000
Phenol-d5	100.00	60.000
1,2-Dichlorobenzene-d4	100.00	60.000
2-Fluorobiphenyl	100.00	60.000
Nitrobenzene-d5	100.00	60.000
p-Terphenyl-d14	100.00	60.000
Multi-Component	100.00	60.000
Aniline	100.00	60.000
Benzoic Acid	100.00	60.000
Benzyl Alcohol	100.00	60.000
Pyridine	100.00	60.000

Parent Std No.: BNA0812-05, CLP SOW OLM04 Semivolatiles — Aliquot Amount (ml): 0.0300
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 02-28-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Acetophenone	2,000.0	60.000
Atrazine	2,000.0	60.000
Benzaldehyde	2,000.0	60.000

Biphenyl	2,000.0	60.000
Caprolactam	2,000.0	60.000

Parent Std No.: BNA0813-05, Equity 8270 ADD-ON Mix Aliquot Amount (ml): 0.0300
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 11-30-2006

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1-Methylnaphthalene	2,000.0	60.000
2,3,4,6-Tetrachlorophenol	2,000.0	60.000
2,3,5,6-Tetrachlorophenol	2,000.0	60.000
2-Naphthylamine	2,000.0	60.000
7,12-Dimethylbenz(a)anthracene	2,000.0	60.000
Methyl methanesulfonate	2,000.0	60.000
N-Nitroso-di-n-butylamine	2,000.0	60.000

Reviewed By: _____

STL Pittsburgh

Standards Preparation Logbook Record

Nov-22-2005

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0810-05, STD160(80ug/ml) 8270/clp/625 — Analyst: bachas

Solvent: Methylene chloride Lot No.: b32e12 Volume (ml): 1.0000
Date Prep./Opened: 10-27-2005
Date Expires(1): 04-27-2006 (6 Months)
Date Expires(2): 11-30-2006 (None)

Parent Std No.: BNA0391-05, 1,4 Dioxane Aliquot Amount (ml): 0.0400
Parent Date Expires(1): 05-20-2006 Parent Date Expires(2): 06-30-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1,4-dioxan	2,000.0	80.000

Parent Std No.: BNA0526-05, EPA 8270 Benzidines Mix Aliquot Amount (ml): 0.0400
Parent Date Expires(1): 07-06-2006 Parent Date Expires(2): 02-28-2008

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
3,3'-Dichlorobenzidine	2,000.0	80.000
3,3'-dimethylbenzidine	2,000.0	80.000
Benzidine	2,000.0	80.000

Parent Std No.: BNA0773-05, 8270/CLP/625 Stock Mix 100ug/ml Aliquot Amount (ml): 0.8000

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
N-Nitrosodiphenylamine	100.00	80.000
2,4,6-Tribromophenol	100.00	80.000
2-Chlorophenol-d4	100.00	80.000
2-Fluorophenol	100.00	80.000
Phenol-d5	100.00	80.000
1,2-Dichlorobenzene-d4	100.00	80.000
2-Fluorobiphenyl	100.00	80.000
Nitrobenzene-d5	100.00	80.000
p-Terphenyl-d14	100.00	80.000
Multi-Component	100.00	80.000
Aniline	100.00	80.000
Benzoic Acid	100.00	80.000
Benzyl Alcohol	100.00	80.000
Pyridine	100.00	80.000

Parent Std No.: BNA0812-05, CLP SOW OLM04 Semivolatiles Aliquot Amount (ml): 0.0400
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 02-28-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Acetophenone	2,000.0	80.000
Atrazine	2,000.0	80.000
Benzaldehyde	2,000.0	80.000

Biphenyl	2,000.0	80.000
Caprolactam	2,000.0	80.000

Parent Std No.: BNA0813-05, Equity 8270 ADD-ON Mix Aliquot Amount (ml): 0.0400
Parent Date Expires(1): 10-27-2006 Parent Date Expires(2): 11-30-2006

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1-Methylnaphthalene	2,000.0	80.000
2,3,4,6-Tetrachlorophenol	2,000.0	80.000
2,3,5,6-Tetrachlorophenol	2,000.0	80.000
2-Naphthylamine	2,000.0	80.000
7,12-Dimethylbenz(a)anthracene	2,000.0	80.000
Methyl methanesulfonate	2,000.0	80.000
N-Nitroso-di-n-butylamine	2,000.0	80.000

Reviewed By: _____

STL Pittsburgh

Standards Preparation Logbook Record

Nov-22-2005

Logbook: \\pitsvr01\stdslog\GCMS Semi-Volatile.std

BNA0811-05, 2nd source verif sstd50(25ug/ml) Analyst: bachas

Solvent: Methylene chloride Lot No.: b32e12 Volume (ml): 1.0000
Date Prep./Opened: 10-27-2005
Date Expires(1): 02-28-2006 (6 Months)

Parent Std No.: BNA0165-05, Equity SS N-Nitrosodiphenylamine Aliquot Amount (ml): 0.0050
Parent Date Expires(1): 03-10-2006 Parent Date Expires(2): 11-30-2006

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
N-Nitrosodiphenylamine	5,000.0	25.000

Parent Std No.: BNA0190-05, Equity SS 8270 Calibration Mix Aliquot Amount (ml): 0.0250
Parent Date Expires(1): 03-31-2006 Parent Date Expires(2): 03-30-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
64 Compounds	1,000.0	25.000

Parent Std No.: BNA0278-05, Equity SS 8270 ADD-ON Aliquot Amount (ml): 0.0125
Parent Date Expires(1): 05-03-2006 Parent Date Expires(2): 07-30-2006

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1-Methylnaphthalene	2,000.0	25.000
2,3,4,6-Tetrachlorophenol	2,000.0	25.000
2,3,5,6-Tetrachlorophenol	2,000.0	25.000
2-Naphthylamine	2,000.0	25.000
7,12-Dimethylbenz(a)anthracene	2,000.0	25.000
Methyl methanesulfonate	2,000.0	25.000
N-Nitroso-di-n-butylamine	2,000.0	25.000

Parent Std No.: BNA0279-05, Equity SS 8270 Benzidines Mix Aliquot Amount (ml): 0.0125
Parent Date Expires(1): 05-03-2006 Parent Date Expires(2): 11-30-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
3,3'-Dichlorobenzidine	2,000.0	25.000
3,3'-Dimethylbenzidine	2,000.0	25.000
Benzidine	2,000.0	25.000

Parent Std No.: BNA0391-05, 1,4 Dioxane Aliquot Amount (ml): 0.0125
Parent Date Expires(1): 05-20-2006 Parent Date Expires(2): 06-30-2007

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
1,4-dioxan	2,000.0	25.000

Parent Std No.: BNA0697-05, Equity SS 8270 Calibration Mix 4 Aliquot Amount (ml): 0.0125
Parent Date Expires(1): 02-28-2006 Parent Date Expires(2): 02-28-2006

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Aniline	2,000.0	25.000
Benzoic Acid	2,000.0	25.000

Benzyl Alcohol	2,000.0	25.000
Pyridine	2,000.0	25.000

Parent Std No.: BNA0782-05, Equity SS CLP OLM04 Semivolatiles Aliquot Amount (ml): 0.0125
Parent Date Expires(1): 10-18-2006 Parent Date Expires(2): 03-30-2008

Component	Initial Conc (ug/ml)	Final Conc (ug/ml)
Acetophenone	2,000.0	25.000
Atrazine	2,000.0	25.000
Benzaldehyde	2,000.0	25.000
Biphenyl	2,000.0	25.000
Caprolactam	2,000.0	25.000

Reviewed By: _____

Appendix B– EPA Memo Regarding Method 625 Modification

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

MEMORANDUM

OFFICE OF WATER

SUBJECT: **Recommended Approved Modifications to EPA Method 625**

FROM: Richard Reding, Chief
Engineering & Analytical Support Branch, EAD, OST

TO: Quality Assurance Managers
ATP Coordinators
NPDES Coordinators

DATE: November 1, 2006

The 304(h) methods branch recommends allowing several modifications to EPA Method 625 for environmental permitting and compliance monitoring under the EPA's Clean Water Act (CWA) programs. This memorandum does not address laboratory certification requirements that states have mandated.

The text in "Protocol for EPA Approval of Alternate Test Procedures for Organic and Inorganic Analytes in Wastewater and Drinking Water" Section 1.3.2 allows flexibility in the modification of "front end techniques" of the test method provided all criteria in this section and **all QC in the method are** met and documented. This protocol can be downloaded at http://www.epa.gov/waterscience/methods.

Recommendations on Method Modifications to EPA Method 625 when Capillary Columns are used:

1. **Combining sample extracts before analysis**

If the analytes can be reliably identified and quantified in the combined extracts, the extracts may be combined. If, however, the identification and quantitation of any analyte is adversely affected by another analyte, a surrogate, or an interferant, the extracts must be analyzed separately. If there is ambiguity, the extracts must be analyzed separately.

2. **Reverse order of pH extraction**

The pH extraction sequence may be reversed to better separate acid and neutral components. Neutral components may be extracted with either acid or base components.

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Previously, neither of these modifications has been used with Method 625 primarily because of limitations of the resolving power of the packed columns used. In 1985, EPA Region 3 Central Regional Lab requested a modification to method 625 as an alternate test procedure (ATP). Although the approval was for limit use by EPA's Region 3, Central Regional Laboratory only, this modification has come to be used throughout the laboratory community (see attached memo).

Why allow these modifications? Following the base-neutral than acid extraction sequence of method 625 in some cases demonstrated the decomposition of some analytes under basic conditions. Organochlorine pesticides may dechlorinate; phthalate esters may exchange; phenols may react to form tannates. These reactions increase with increasing pH. Reversing the extraction pH sequence may better separate acid and neutral waste components.

Other Recommended Modifications to Method 625

A smaller sample volume may be used to minimize matrix interferences provided matrix interferences are demonstrated and documented.

Alternate surrogate and internal standard concentrations other than those specified in the method are acceptable provided that method performance is not degraded;

An alternate calibration curve and a calibration check other than those specified in the method;

A different solvent for the calibration standards to match the solvent of the final extract.

Other Method Flexibility News

We are revising the "Guidance on Evaluation, Resolution, and Documentation of Analytical Problems Associated with Compliance Monitoring" often referred to as the "Pumpkin Book". Many of the recommendations in the revised "Pumpkin Book" cover ways to mitigate matrix effects.

More explicit flexibility to make changes in approved methods without prior EPA approval is now described at 40 CFR Part 136.6. Such changes are only allowed if the modified method produces equivalent performance for the analyte(s) of interest, and the equivalent performance is documented. It is essential to consult the full text at 40 CFR 136.6 before undertaking method modifications.

Please feel free to forward this information. If you have any questions regarding this memorandum, please contact Lemuel Walker of EASB/EAD/OST by email at walker.lemuel@epa.gov.

cc Lemuel Walker
ATP Coordinator

Appenidx C

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
Breakdown Check 8081A: Endrin, DDT 8270C: DDT	Analysis of s standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degradation of either DDT or Endrin exceeds method–specified criteria, corrective action must be taken before proceeding with calibration.
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified.	To verify the identification of an analyte.	This is a required QC procedure. All positive results must be confirmed.
CCV	This verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.	To verify that instrument response is reliable, and has not changed significantly from the current ICAL.	If the values for the analytes are outside the acceptance criteria, the initial calibration may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging.
Demonstrate Acceptable Analyst Capability	Analyst runs QC samples in series to establish his/her ability to produce data of acceptable accuracy and precision.	To establish the analysts' ability to produce data of acceptable accuracy and precision.	The average recovery and standard deviation of the replicate must be within designated acceptance criteria.
Duplicate Sample	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are taken and prepared and analyzed identically.	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix.	To provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the sample and the duplicate.
ICAL	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method.	To establish a calibration curve for the quantification of the analytes of interest.	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be

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DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
			analyzed.
Internal Standards	A known amount of standard added to all standards and samples as a reference for evaluating and controlling the precision and bias of the applied analytical method.	To verify that the analytical system is in control.	Any sample associated with out-of-control results must be reanalyzed.
LCS containing all analytes required to be reported	A QC standard of known composition prepared using reagent free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern.	To evaluate method performance by assessing the ability of the lab/analyst to successfully recover the target analytes from a control (clean) matrix.	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the accuracy/bias of the measurement system.
MS	A sample prepared by adding a know amount of targeted analyte(s) to an aliquot of a specific environmental sample.	To assess the performance of the method as applied to a particular matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty.
MSD	A 2nd replicate MS prepared in the lab, spiked with an identical, known amount of targeted analyte(s), and analyzed to obtain a measure of the precision of the recovery for each analyte.	To assess the performance of the method as applied to a particular matrix and provide information of the homogeneity of the matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix.
MDL Verification Check	A low-level spike taken through the prep and analytical steps at approximately 2x the MDL used to verify that the laboratory can detect analytes at the calculated MDL.	To validate the MDL on an ongoing basis	If the MDL verification check fails, reprep/reanalyze at a higher level to set a higher MDL or the MDL study must be repeated.
MB	A sample of a matrix similar to the batch of associated samples in which no target analytes or interferences are present at concentrations that impact the analytical	To assess background interferences or contamination in the analytical system that might lead to high bias or false	This QC is used to measure lab accuracy/bias. The MB could indicate whether contamination is occurring during sample prep and analysis. If

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DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
	results.	positive data.	analytes are detected > ½ RL, reanalyze or B-Flag results for all samples in prep batch. For common lab contaminants, no analytes detected > RL. See DoD Box D-5; & Sec. D.1.1.1
MDL Study	The process to determine the minimum concentration of an analyte 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.	To determine the lowest concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.	MDLs must be established prior to sample analysis. The RL or LOQ is at least 3x the MDL. Used in combination with the MDL verification check to validate the MDL on an ongoing basis.
RT window position establishment for each analyte (chromatographic methods only)	Determination of the placement of the RT window (start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the initial calibration.	To identify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, and/or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
RT window verification for each analyte (chromatographic methods only)	A standard is used to verify that the width and position of the RT windows are valid so that accurate analyte identification can be made during sample analysis.	To minimize the occurrence of both false positive and false negative results at each calibration verification.	The peaks from the standard used are compared to the RT window established during the ICAL to verify that the analytes of interest still fall within the window.
Second source calibration verification	A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration.	To verify the accuracy of the initial calibration.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared to the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.
Surrogate spike (organic analysis only)	A pure substance with properties that mimic the analyte of interest. Surrogates are compounds unlikely to be found in	To assess the ability of the method to successfully recover specific non-target analytes	Whereas the MS is normally done on a batch-specific basis, the surrogate spike is done on a sample-specific

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DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
	environmental samples to evaluate analytical efficiency by measuring their % Recovery.	from an actual matrix.	basis. Taken with the information derived from other spikes (LCS; MS), the bias in the analytical system can be determined.
Tuning (MS methods only)	The analysis of a standard compound to verify the mass spectrometer meets standard mass spectra abundance criteria prior to sample analysis.	To verify the proper working of the mass spectrometer.	Proper tuning of the mass spectrometer must be verified prior to sample analysis .

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.

2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

3. If the requirements in the DoD tables do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3: Organic Analysis by GC/MS - Methods 8260 and 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
IDOC	Per Instrument/Analyst	DoD acceptance criteria if available; otherwise method specific criteria.	Correct / Repeat for those analytes which failed criteria.	NA
MDL	Annually or quarterly MDL Checks performed	40 CFR 136B; MDL verification checks must produce a signal at least 3x the instrument's noise level.	Run MDL check at higher level and set MDL higher or reconduct MDL study.	NA
Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method specific ion criteria.	Retune instrument and verify. Rerun affected samples.	NA
Breakdown check DDT (8270C only)	Daily prior to analysis of samples	Degradation $\leq$ 20% for DDT (Benzidine & PCP should be present at their normal response and no peak tailing should be observed).	Correct problem then repeat breakdown check.	NA
ICAL	Initial 5-point calibration prior to sample analysis	1. Average RF for SPCCs: VOCs - $\geq$ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, $\geq$ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs - $\geq$ 0.050 2. RSD for RFs for CCCs: VOCs and SVOCs - $\leq$ 30% and one option below. **Option 1:** RSD for each analyte $\leq$ 15% **Option 2:** linear least squares regression: r $\geq$ 0.995 **Option 3:** non-linear regression: Coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for 2nd order, 7 points shall be used for 3rd order)	Correct problem then repeat initial calibration.	NA
2nd Source	Once after each	Value of 2nd source for all analytes	Correct problem and verify 2nd	NA

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3: Organic Analysis by GC/MS - Methods 8260 and 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
calibration verification	initial calibration	within ± 25% of expected value - See SOP Section 10.5.15 for exception an DoD SOP.	source standard. Rerun, if that fails, correct problem and repeat ICAL.	
RT window position establishment for each analyte	Once per ICAL	Position shall be set using midpoint standard of the initial calibration curve.	NA	NA
Evaluation of Relative RT (RRT)	With each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct problem, then rerun ICAL	NA
Calibration verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time	1. Average RF for SPCCs: VOCs - ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs - ≥ 0.050 2. %Difference for CCCs: VOCs and SVOCs - ≤ 20% D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration) All calibration analytes must be within 20% D, with no individual analytes (except CCC's) > 25% D (DoD Version 2.2)	Correct problem, rerun CV, if fails, repeat ICAL (Data associated with an unacceptable CCV may be fully usable under the following conditions: 1. CCV (high bias) and samples ND, then raw data may be reported with appropriate flag 2. CCV (low bias) and samples exceed maximum regulatory limit/decision level (DoD Box 60: Project specific permission from appropriate DoD personnel is required to report data generated from a run with noncompliant CCV.)	Apply J-flag to all results associated with the analytical batch for all analytes > 20%D and < 25% D. Identify in case narrative analytes > 20% D. (DoD Version 2.2) Apply Q-flag if no sample material remains and analyte exceeds criteria
Internal Standards verification	In all field samples and standards	RT ± 30 seconds from RT of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Apply Q-flag to analytes associated with the non-compliant IS.
MB	One per prep batch	No analytes detected > ½ RL For common lab contaminants, no analytes > RL	Correct problem, then see criteria in box D-5; if required, reprep/reanalyze MB and all associated samples.	Apply B-flag to all results for the contaminated analyte for all samples in the

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3: Organic Analysis by GC/MS - Methods 8260 and 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
				associated prep batch.
LCS (containing all analytes to be reported)	One LCS per prep batch	DoD specified QC criteria, if available	Correct problem, reprep/reanalyze the LCS and all samples in the associated prep batch for all failed analytes, if sufficient sample is available.	Apply Q-flag to specific analyte(s) in all samples in the prep batch.
MS	One per prep batch per matrix	For matrix evaluation, use DoD specified QC criteria for LCS.	Examine the project-specific DQOs. Contact client for additional corrective action measures.	Apply J-flag to specific analyte(s) in the parent sample.
MSD or Sample Duplicate	One per prep batch per matrix	RPD $\leq$ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact client for additional corrective action measures.	Apply J-flag to specific analyte(s) in the parent sample.
Surrogate	All field and QC samples	DoD specified QC criteria if available, otherwise method specific criteria or lab's own in-house criteria.	For QC and field samples, correct problem, reprep/reanalyze all failed samples in the associated prep batch if sufficient sample material is available.	Apply J-flag for specific analyte(s) in all field samples collected from the same site matrix as the parent. Apply Q-flag to QC samples for specific analyte(s)
Results reported between LOD and LOQ			Apply J-flag to all results between LOD (MDL) and LOQ (RL)	
Manual Integration	When manual integrations are performed	Raw data shall include a complete audit trail for those manipulations, raw data output showing the results of the MI (i.e., chromatograms of manually integrated peaks), and notation of rationale, date, and signature/initials of person performing manual operation.		Apply M-flag to MI data

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-

specific direction based on DQOs is not available.

2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

3. If the requirements in the DoD tables do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

THIS IS A CONTROLLED DOCUMENT. WHEN PRINTED IT BECOMES UNCONTROLLED.

Title: DETERMINATION OF VOLATILE ORGANICS BY GC/MS

[SW-846 Method 8260B AND EPA METHOD 624]

Approvals (Signature/Date):

09/11/07	09/11/07
Sharon Bacha Date	Larry Matko Date
Technical Manager	Acting Health & Safety Manager
09/11/07	09/11/07
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Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. PITT-MS-0002, Rev 9.

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APPENDIX A

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Table A-1 Method 624 Analytes and Reporting Limits

Table A-2 Method 624 QC Acceptance Criteria

Table B-1 DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3 DoD QSM Version3: Requirements for Organic Analysis by GC/MS - Methods 8260 and 8270

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges and other solid matrices. Standard analytes are listed in Tables 1, 2, and A-1.

1.2. This SOP is applicable to method 8260B and 624. Appendix A present modifications to the procedures in the main SOP that are necessary for analysis of water samples by method 624. For DoD requirements refer to SOP PITT-QA-DoD-0001, Implementation of DoD QSM Version 3, January 2006.

1.3. This method can be used to quantify 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, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 200 µg/L for 5 mL standard level waters, 1 to 40 µg/L for low level waters, 5 to 200 µg/kg for low-level soils, and 250 to 25,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1, 2, and A-1.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2. SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be frozen and purged directly.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and back flushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples, and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3. DEFINITIONS

3.1. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will normally start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is received, an LCS/LCSD will be used in the place of an MS/MSD. Refer to the TestAmerica Pittsburgh QC Program document (QA-003) for further details of the batch definition.

3.2. Method Blank

A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

3.3. Laboratory Control Sample (LCS)

Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

3.4. Surrogates

Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC)

CCCs are a representative group of compounds, which are used to evaluate initial calibrations and continuing calibrations. Relative standard deviation (%RSD) for the initial calibration and % drift or % deviation (%D) for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

SPCCs are compounds, which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. Response factors from the initial and continuing calibrations are calculated for the SPCC compounds and compared to the specified method criteria.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered an antifoaming agent (Dow Corning Antifoam C) can be used. A blank spiked with this agent must be analyzed with the sample to show there is no target interferences induced by this agent. The antifoaming agent is only used on one particular client's TCLP samples. The antifoaming agent is not used routinely. If it needs to be used, approval from Project Manager is obtained.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2. 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.

5.3. 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.

5.4. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.5. 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.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.6. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately.

5.7. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred,

and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.8. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.9. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 uL and larger, 0.006 inch ID needle.

6.2 Syringe: 5 or 25 mL glass with luerlok tip, if applicable to the purging device.

6.2. Balance: Top-loading balance capable of weighing 0.01 g

6.3. Glassware:

6.3.1. Vials: 40 mL with screw caps and Teflon liners.

6.3.2. Volumetric flasks: 10 mL, 50 mL and 100 mL, class A with ground-glass stoppers.

6.4. Spatula: Stainless steel.

6.5. Disposable pipettes: Pasteur.

6.6. pH paper: Narrow range.

6.7. Gases:

Helium: Ultra high purity, gr. 99.999%.

6.8. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.8.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm

at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.8.2. Trap: OI # 10

6.8.3. Desorber: The desorber should be capable of rapidly heating the trap to at least 180°C. Many such devices are commercially available.

6.8.4. Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low level soil analysis.

6.9 Gas Chromatograph/Mass Spectrometer System:

6.9.1 Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.9.2 Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.9.2.1 Column 1: 20m x 0.18 ID J&W DB-624 or Restek 502.2 with 1 µm film thickness.

6.9.3 Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng or 25 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.9.4 Data System: A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference

library. The computer system must also be capable of backing up data for long-term off-line storage.

7 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Methanol: Purge and Trap Grade, High Purity

7.1.2 Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.5) Reagent water is obtained from Millipore system. Other methods of preparing reagent water are acceptable.

7.1.3 1:1 HCl

7.1.4 10% Sodium thiosulfate, (ACS) Granular

7.2 Standards

7.2.1 Calibration Standard

7.2.1.1 Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10° to -20°C.

7.2.1.2 Working standards: A working solution containing the compounds of interest is prepared from the stock solution(s) in methanol. The working standard solutions will be prepared monthly with the exceptions of the gases and 2-chloroethylvinyl ether solutions, which will be prepared on a weekly basis. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20% then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

7.2.1.3 Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4 If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers expiration date.

7.2.2 Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 6 for internal standard components.

7.2.3 Surrogate Standards: Refer to Table 7 for surrogate standard components and spiking levels.

7.2.4 Laboratory Control Sample Spiking Solutions: Refer to Table 8 for the normal control LCS components and spiking levels.

7.2.5 Matrix Spiking Solutions: The matrix spike contains the same control components as the LCS. Refer to Table 8.

7.2.6 Tuning Standard: A standard is made up that will deliver up to 50 ng on column upon injection. A recommended concentration of 25 ng/µL of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

8 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Holding time for preserved volatile samples is 14 days from sample collection. Holding times for unpreserved waters is 7 days. Holding time for unpreserved soils requires that they are analyzed or preserved within 48 hours of sampling.

8.2 Water samples are normally preserved at pH $\leq$ 2 with 1:1 hydrochloric acid.

8.3 Several different approaches to sample preservation and storage are presented below. The appropriate procedure selection is subject to project or program specific requirements.

8.4 Solid samples are prepped in a VOA vial with volatile free water and frozen within 48 hours of sampling for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted. Terra Core™ kits (from C &G Scientific) can also be used. The kits are shipped

to the field. Each kit includes two low level vials, one medium level vial and one bottle for percent moisture. One kit is used per each sample.

8.5 There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore sample. (The 5 g or 25 g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore samplers or to use field preservation. The water preservation with freezing method is referenced in Method 5035A, Sec 8.2.1.2 and Appendix A table A-1.

8.6 Sample collection for medium level analysis using EnCore samplers.

8.6.1 Ship one 5 g (or 25 g) EnCore sampler per field sample position.

8.6.2 An additional bottle must be shipped for percent moisture determination.

8.7 When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label. The surrogate and the matrix spike solution is added at the time of analysis.

8.7.1 Prepare an LCS for each batch. Spike the LCS at the time of analysis.

8.7.2 Shake the samples for two minutes to distribute the methanol throughout the soil.

8.7.3 Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4 ± 2 °C until analysis.

8.8 Sample collection for medium level analysis using field methanol preservation

8.8.1 Prepare a 2 oz sample container by adding 25 mL purge and trap grade methanol. (If a 5 g sample is to be used, add 5 mL methanol to a VOA vial. The surrogate and matrix spike solution is added at the time of analysis).

8.8.2 Seal the bottle and attach a label.

8.8.3 Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.8.4 Ship with appropriate sampling instructions.

8.8.5 Each sample will require an additional bottle with no preservative for percent moisture determination.

8.8.6 At client request, the methanol addition and weighing may also be performed in the field.

8.8.7 When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.9 Low level procedure

8.9.1 If low detection limits are required (typically < 50 µg/kg) freezing the EnCore may be used. However, it is also necessary to take a sample for the medium level (field methanol preserved or using the EnCore or Terra Core™ sampler) procedure, in case the concentration of analytes in the soil is above the calibration range of the low level procedure. (Note: OVAP samples cannot be frozen.)

8.9.2 A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.9.3 The soil sample is taken using a 5g EnCore sampling device and returned to the lab. It is recommended that two EnCore samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.9.4 Prepare VOA vials by adding 5 mL of reagent water only.

8.9.5 Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.9.6 Weigh the vial to the nearest 0.01g and note the weight on the label.

8.9.7 Extrude the soil sample from the EnCore sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note. Water preserved vials must be frozen.

8.9.8 Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative

for percent moisture determination, and an additional bottle preserved with methanol for the medium level procedure. Depending on the type of soil it may also be necessary to ship vials with no or extra preservative.

8.10 Unpreserved soils

8.10.1 At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on SW-846 Method 5030A. It is no longer included in SW-846 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.10.2 For OVAP samples the 5030A approach may only be used for samples that are > 200 ppb.

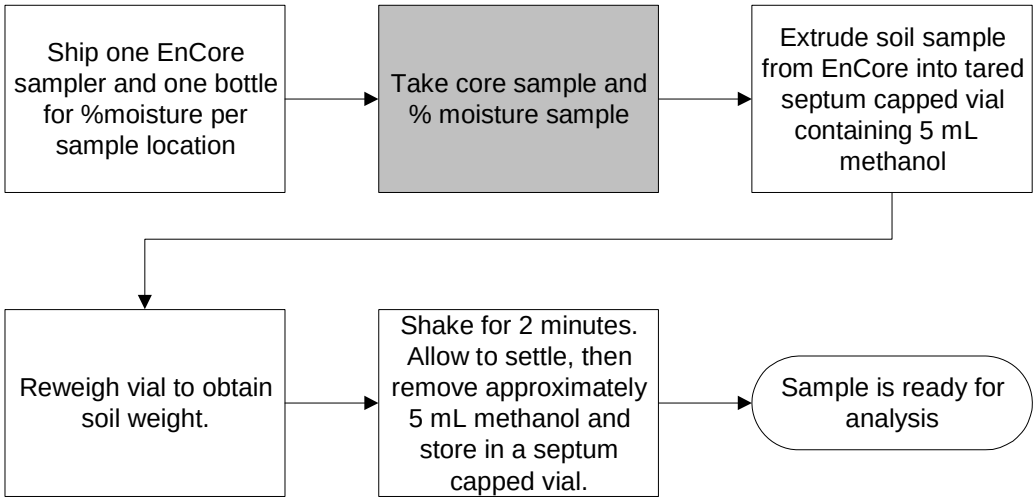
8.11 Aqueous samples are stored in glass containers with Teflon lined septa at $4^{o}C \pm 2^{o}C$, with minimum headspace.

8.12 Medium level solid extracts are aliquoted into 2 mL glass vials with Teflon lined caps and stored at $4^{o}C \pm 2^{o}C$. The extracts are stored with minimum headspace.

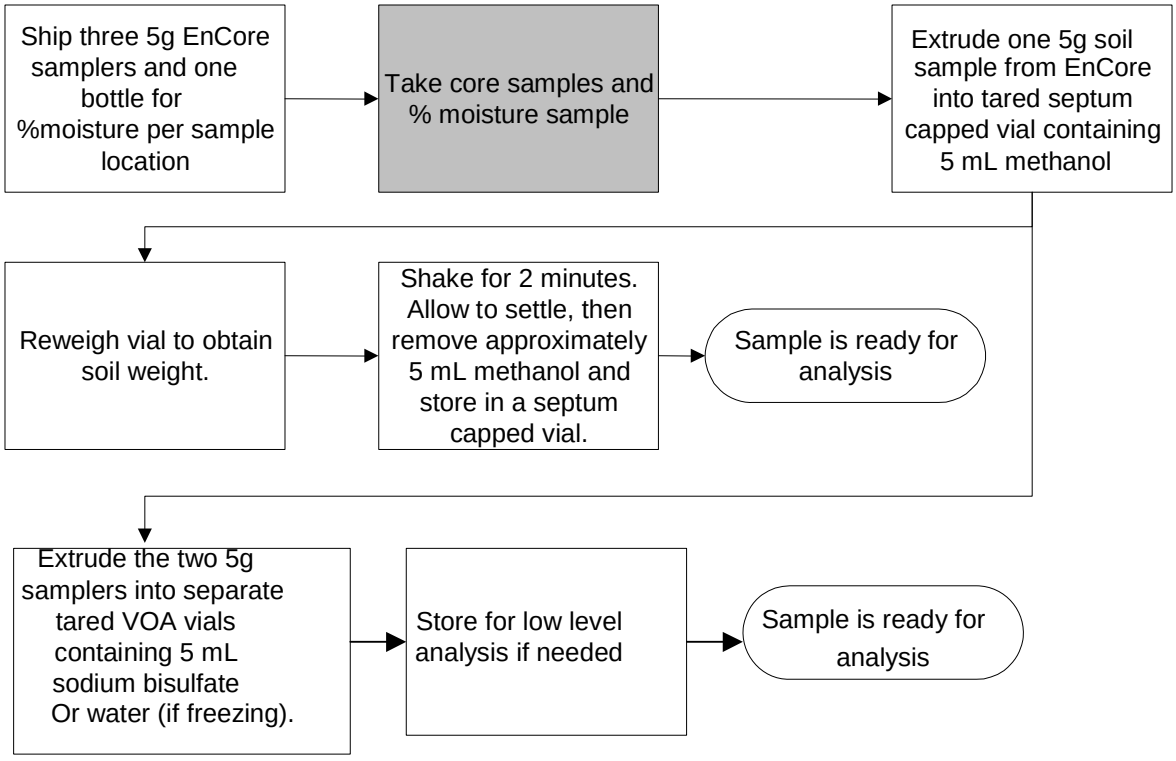
8.13 The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore sampler (before the sample is added to methanol or frozen) is 48 hours.

8.14 A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 7 days.

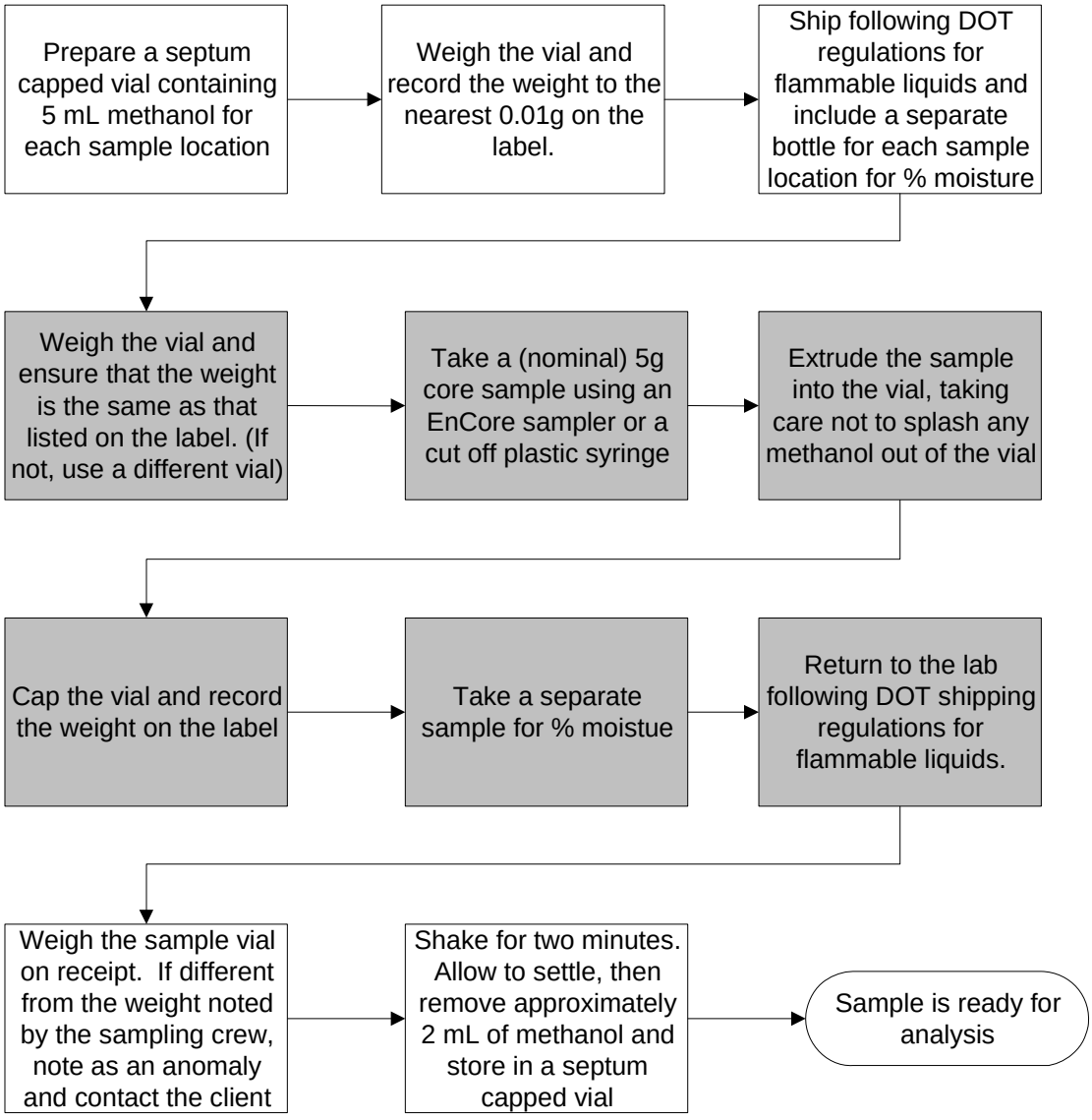
EnCore procedure when low level is not required (field steps in gray)



EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



9 QUALITY CONTROL

9.1 See Document QA-003 "TestAmerica Quality Control Program" for additional detail. For DoD requirements refer to SOP # PITT-QA-DoD-0001, Implementation of DoD QSM Version 3 January 2006, current version and DoD Tables B-1 and B-3.

9.2 Initial Demonstration of Capability

9.2.1 Section 13 and method detection limit (MDL) studies must be acceptable before analysis of samples may begin. MDLs should be analyzed for low and medium soils and aqueous samples. MDLs for the analytes of interest are performed annually.

9.2.2 For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of a standard at the reporting limit and a single point calibration.

9.2.3 IDOC is performed for each new analyst and method. Four LCS are processed through the method. The QC criteria is listed in Table 13.

9.3 In-house historical control limits have been determined for surrogates, matrix spikes, and laboratory control samples (LCS). The LCS limits for method 624 are defined in the method and are listed on Table A-2. These limits must be re-checked at least annually. The recovery limits are mean recovery ± 3 standard deviations for surrogates, matrix spikes and LCS. Precision limits for matrix spikes / matrix spike duplicates are 0 to mean relative percent difference ± 3 standard deviations.

9.3.1 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3.2 Refer to the QC Program document (QA-003) for further details of control limits.

9.4 Surrogates

Every sample, blank and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 8. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions)

9.4.1 Check all calculations for error.

9.4.2 Ensure that instrument performance is acceptable.

9.4.3 Recalculate the data and/or reanalyze if either of the above checks reveal a problem

9.4.4 Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem

9.4.5 Samples that have major matrix interference, which is obvious from the chromatogram, will not be rerun for confirmation of matrix interference.

9.4.6 The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.4.7 If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.4.8 Refer to the TestAmerica Pittsburgh QC Program document (QA-003) for further details of the corrective actions.

9.5 Method Blank

For DoD method blank criteria, see SOP # PITT-QA-DoD-0001. For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. If the first method blank does not meet criteria, a second blank may be analyzed. The method blank must meet criteria before proceeding with sample analyses. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of 100 ul of methanol extract into 4.9 mls of reagent water. Surrogates are added and the method

blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

9.5.1 If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone) the data may be reported with qualifiers if the concentration of the analyte is not more than five times the reporting limit. Such action must be taken in consultation with the client.

9.5.2 Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

9.5.3 If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.5.4 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the sample analysis is free of contamination. All non-conforming blanks will be documented in a non-conformance memo and if reported the reasons for reporting the data will be summarized. For example, if surrogate recoveries are low, re-extraction and/or reanalysis of the blank and affected samples will normally be required. Consultation with the client should take place. If the surrogate recoveries are high and there are target compounds found in the associated sample the samples will require re-extraction and/or reanalysis.

9.5.5 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all compounds detected in the blank are flagged with a "B" in the associated samples, and appropriate comments are made in a narrative to provide further documentation.

9.5.6 Refer to the TestAmerica Pittsburgh QC Program document (QA-003) for further details of the corrective actions.

9.6 Laboratory Control Samples (LCS)

For DoD LCS criteria, see SOP # PITT-QA-DoD-0001. For each batch of samples, analyze a LCS. The LCS is analyzed after the calibration standard. The LCS contains a representative subset of the analytes of interest (See Table 8), and must contain the same

analytes as the matrix spike. If any control analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch. Please refer to Appendix A and Table A-2 for LCS criteria for method 624.

9.6.1 If the batch cannot be re-prepped and/or reanalyzed due to insufficient sample, a discussion should be provided of the data quality indicators and must be clearly presented in the project records and the report.

9.6.2 If re-extraction and/or reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6.3 Refer to the TestAmerica Pittsburgh QC Program document (QA-003) for further details of the corrective action.

9.6.4 If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client. Unless otherwise agreed only the control analytes (Table 8) are used to evaluate analytical performance control.

9.7 Matrix Spikes

For DoD MS/MSD criteria, see SOP # PITT-QA-DoD-0001. For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 8. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits. Refer to Table A-2 for method 624 spike limits.

9.7.1 If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

9.7.2 If the recovery for any control component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory operation is out of control and

corrective action must be taken. Corrective action will normally include reanalysis of the batch.

9.7.3 If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

9.7.4 The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.8 Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.9 Quality Assurance Summaries

Certain clients may require specific project or program QC, which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.10 TestAmerica Pittsburgh QC Program

Further details of QC and corrective action guidelines are presented in the TestAmerica Pittsburgh QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10 CALIBRATION AND STANDARDIZATION

10.1 Summary

Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of 4-Bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of six concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system. The use of separate calibrations is required for water and low soil matrices.

10.2 Recommended Instrument Conditions

10.2.1 General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	to give at least 5 scans/peak, but not to exceed 2 second/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 15 mL/minute
Make-up Gas Flow:	25–30 mL/minute

10.2.2 Gas chromatograph suggested temperature program

Parameter	Sample Analysis	BFB Analysis
Initial Temperature:	35°C	35°C
Initial Hold Time:	4 minutes	2 min
Temperature Program:	15°C/minute	20°C/minute
Final Temperature:	200°C	200°C
Final Hold Time:	1.1 minutes	1.0 min.

10.3 Instrument Tuning

Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 9 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB

10.3.1 Acceptable procedures for BFB tuning are as follows:

10.3.1.1 The peak apex, or the scan immediately before the apex, or the scan immediately after the apex, or the average of these three scans may be used. The average of the apex and the scan before or after the apex may also be used.

10.3.1.2 Background subtraction is optional. If background subtraction is used, a single scan must be subtracted. This single scan must be prior to and within 20 scans of the start of BFB elution but must not be part of the BFB peak.

10.3.1.3 If the instrument has a built in macro that checks the BFB, use of this macro with no manual manipulation is also acceptable. (Assuming, of course that the correct ion ratios are being checked.)

10.3.1.4 NOTE: If the background scan selected includes significant ions at 95 or 174 or 176, then the scan is almost certainly part of the BFB peak and is not acceptable.

10.4 Initial Calibration

10.4.1 A series of six initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Typical calibration levels for a standard 5 mL purge are: 5, 25, 50, 100, 200 and 250 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Typical calibration levels for a Low Level purge are 1, 5, 10, 15, 20, and 40 µg/L. Again, some analytes are prepared at higher levels. Tables 3 and 4 list the calibration levels for each analyte. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit. See Table 3 and 4 for medium level soil standard concentration.

10.4.2 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 3) and the Appendix IX standard (Table 4). If acceptable analytical performance can be obtained the primary and appendix IX standards may be analyzed together.

10.4.3 Internal standard calibration is used. The internal standards are listed in Table 6. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See equation 1, Section 12, for calculation of response factor.

10.4.4 The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 11 for the CCCs. Acceptable CCC compounds will use average RF curve.

10.4.4.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.5 The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 10 for the SPCC compounds and required minimum response factors.

10.4.6 Note: the laboratory may not use the "grand mean" rule. The following are guidelines that are used for routine SW-846 analysis within the laboratory, however these guidelines are subject to program and project specific requirements.

10.4.6.1 Where a target compound is ≤15% RSD an average response factor curve may be used. If the 15% RSD criteria are exceeded the analyst must assess the curve and attempt to apply a "best-fit" curve function and a graphical representation of the curve will be provided as documentation of this review. The first step of the assessment is to find out if the quadratic curve will have a correlation coefficient of ≤ .995. If it does not, then use the average response factor. If it does, then review where the quadratic curve intercepts the y-axis in comparison to the MDL and origin. Also review the shape of the curve. Does it overlap itself or have other potential problems? These steps should all be used in deciding when a quadratic curve or average response factor curve would be best.

10.4.6.2 Where a quadratic or polynomial curve is used R must be ≥.995 for a curve to be considered to be an acceptable fit.

10.4.6.3 All linear curves for non-CCC compounds that exceed 15% RSD or best-fit curve functions that have R < .995 are in exceedance of guidance criteria and must be evaluated for corrective action. The following exceptions may be reportable with narration depending on the project DQO's and data usability requirements:

10.4.6.4 Where a target compound is ≥15% but ≤30% an average response factor curve may still be used if the analyst shows that the average response factor is an acceptable fit over the range of use. A graphical representation of the curve should be presented for documentation. However, if the quadratic curve is clearly a better fit it should be used.

10.4.6.5 Compound list will be divided into two lists: List 1 (reliable performers) and List 2 (poor performers). List 1 compounds should always have a %RSD less than 30% or correlation coefficient of .995 with an allowance of up to two sporadic marginal failures for volatiles. Sporadic marginal failures for these compounds should be </= 40% or .990. Sporadic marginal failures require a print out of the curve and narration.

10.4.6.6 List 2 compounds is comprised of the list of known poor performers. For List 2 analytes, where the %RSD is ≤15% an average response factor will be used. For %RSDs >15% and ≤60% the best fit curve will be selected. For these compounds a print out of the curve will be provided as a graphical documentation of curve performance.

10.4.6.7 Documentation: Raw target curve summary with all compounds set to average response factor will be provided. If quadratic or polynomial equations are used a reprint of the curve table will be provided to show the correlation coefficient for the "best fit" equation. And as noted above, compounds that need additional documentation to demonstrate the curve fit will have a graphical presentation of the curve provided for reference.

10.4.6.8 Any analyte not on List 1 or List 2 would be held to specific criteria based on project specific requirements.

10.4.6.9 Any non-CCC compound being reported from a curve that does not meet either the 15% RSD criteria or the R = .995 for a "best-fit" curve will be narrated as a non-conformance.

10.4.6.10 All %RSDs that are >30% must be narrated and when using an average response factor curve for a %RSD >30% this should also be narrated.

10.4.6.11 Note: Project Specific DQOs or program specific requirements supercede routine lab reporting practices listed in this section.

10.4.7 Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.8 If time remains in the 12-hour period initiated by the BFB injection after the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9 A separate six-point calibration must be prepared for analysis of low level soils. Low level soil analyses require the use of a closed vial autosampler, such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1 g sodium bisulfate, if using sodium bisulfate preservation or 5ml of water if freezing. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Medium soil extracts should be analyzed using the water (unheated) calibration curve.

10.4.10 Non-standard analytes are sometimes requested. Where it is acceptable to the client, it may be is possible to analyze a single standard at the reporting limit (to screen for the compounds) with each continuing calibration rather than a six point initial calibration. If the analyte is detected in any of the samples, a six point initial calibration must be generated and the sample(s) reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary. This is not an acceptable procedure for compliance work. When doing non-standard analytes an MDL will be run before analysis.

10.4.11 All ICALs will be verified by a Second Source Standard. The acceptance criteria will be 80-120% for most compounds and 50-150% for poor method performers. The poor performers are footnoted in Tables 3 and 4. Any compound not listed will fall into the 50-150% criteria until knowledge of the compound can be developed. For DoD second source must be ± 25% with exceptions, refer to SOP PITT-QA-DoD-0001.

10.4.12 Outliers will be evaluated on a project by project basis and narrated in the case narrative if necessary.

10.5 Continuing Calibration: The initial calibration must be verified every twelve hours.

10.5.1 Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 3 calibration standard is used as the continuing calibration.

10.5.2 The RF data from the continuing calibration standards are compared with the average RF from the initial five-point calibration to determine the percent drift or percent deviation of the CCC compounds. The calculations are given in equations 4 (Section 12.3.4) and equation 5 (Section 12.3.5).

10.5.3 Continuing Calibration Verification

10.5.3.1 Calculation Type

10.5.3.1.1 Average Response Factor curves should be verified using a %Difference equation. The %Difference equation compares the RRF factor calculated for the Calibration Verification Standard to the Average RRF of the curve.

10.5.3.1.2 The Quadratic Curves should be verified using a %Drift equation. The %Drift equation compares the measured value of the Calibration Verification Standard to the theoretical value of the standard..

10.5.3.2 %Difference and %Drift Criteria

10.5.3.2.1 CCCs must be ≤20 %Diff

10.5.3.2.2 List One compounds that are non-CCCs must be ≤25 %Diff or Drift

10.5.3.2.3 Up to two Volatile and four Semivolatile compounds that are List One analytes may exceed the 25% criteria, but must be ≤40%.

10.5.3.2.4 List Two Target Analytes including Appendix IX compounds will be accepted where the % Difference or % Drift ≤50%. Please see Table 4-1.

10.5.3.2.5 Where a CCV is out high by >50% and the compound is ND in the samples, the samples may be reported with narration.

10.5.3.3 RRF Criteria

10.5.3.3.1 SPCCs must be as per method requirements. Please see table 10.

10.5.3.3.2 All other compounds must be ≤0.01 (footnote exceptions).

10.5.4 If the CCCs and/or the SPCCs do not meet the criteria in Sections 10.5.3 after the continuing calibration has been attempted twice, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration.

10.5.5 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample desorbed less than or equal to 12 hours after the BFB is acceptable.)

11 PROCEDURE

11.1 Procedural Variations

11.1.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.1.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.1.3 See Appendix A for method 624 criteria.

11.2 Preliminary Evaluation

11.2.1 Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.2.2 Samples are screened on a headspace analyzer. The instrument is calibrated for select compounds at three levels. There are 200ppb, 500ppb, and 1000ppb. 5 mLs of sample are then analyzed on the headspace analyzer and the results are used to calculate a dilution, if necessary, for the sample.

11.2.3 Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 5 µL of sample then serial dilutions must be made in volumetric flasks.

11.2.3.1 The diluted concentration is to be estimated to be in the upper half of the calibration range. The upper range will be defined as the 4th calibration point and above.

11.3 Sample Analysis Procedure

11.3.1 All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2 All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD, a LCS, and a method blank.

11.3.2.1 If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next 12 hour tune period. However, if any instrument corrective action is required, or if a period of greater than 12 hours (SW-8260B) from the preceding BFB tune has passed, a new batch must be started. In other words a QC batch may be kept open for two adjacent and uninterrupted tune periods where both

pass all BFB, CCAL, blank and LCS criteria up to a maximum of 24 hours. For medium level soils the batch is defined at the sample preparation stage. For method 624 the batch tune period is 24 hours.

11.3.2.2 Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.3.2.3 It is not necessary to reanalyze batch QC with the reanalyses of samples. However, any reruns must be as part of a valid batch.

11.3.3 For manual integration practices refer to TestAmerica corporate SOP, S-Q-004, Acceptable Manual Integration Practices. For DoD and all other projects the following criteria must be met:

When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of manual integration (i.e., chromatograms of manually integrated peaks), and notation of rationale, date, and name or initials of person performing manual integration operation (electronic signature is acceptable). DoD QSM, Version 3, Clarification 50 and 57.

Case Narrative. For DoD the case narrative shall provide: identification of **samples and analytes** for which manual integration was necessary. DoD QSM, Version 3, Appendix DoD-A.

11.3.4 Retention time criteria for samples

Retention time windows must be established and verified once per ICAL and at the beginning of the analytical shift as per DoD QSM, Version 3, Appendix DoD-B, Table B-3. If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.3.4.1 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
Retention Time window position establishment for each analyte and surrogate	Once per ICAL	Position shall l be set using the midpoint standard of the initial calibration curve.	NA	NA
Evaluation of relative retention times (RRT)	With each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.

11.4 Water Samples

11.4.1 All samples and standard solutions must be at ambient temperature before analysis.

11.4.2 Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is ≥ 5 µL. Check and document the pH of the remaining sample.

11.4.3 Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution, refer to Tables 6 and 7). The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 50 µg/L solution for a standard 5 mL sample, and a 10 µg/L solution for low level analyses, when added to a 25 mL sample aliquot). Inject the sample into the purging chamber. Note: Low level analyses on instruments that sample directly from the VOA vial (i.e., Archons) use a 5 ml sample volume. Therefore, 1.0 µL of a 250 µg/mL solution of internal standards and surrogates are added to the sample for the regular 5 mL waters and 1uL of a 50 ug/mL solution is added for low level waters.

11.4.3.1 For TCLP samples use 0.5 mL of TCLP leachate with 4.5 mL reagent water and spike with 10 µL of the 25 µg/mL spiking solution. (Note that TCLP reporting limits will be 10 times higher than the corresponding aqueous limits).

11.4.4 Purge the sample for eleven minutes (the trap must be ≤ 35°C).

11.4.5 After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to

condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.4.6 Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.5 Methanol Extracted Soils

11.5.1 Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add 100 µL for a 5 mL purge methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard. Load the sample onto the purge and trap device and analyze the same as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

11.6 Liquid wastes that are soluble in methanol and insoluble in water.

11.6.1 Pipette 1 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.01 gram.

11.6.2 Quickly add 8 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD, 7 mL of methanol, 1 mL of surrogate solution, and 1 mL of matrix spike solution is used.

11.6.3 Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add 100 µL for a 5 mL purge methanolic extract (from Section 11.6.2) to the syringe. Add internal standard. Load the sample onto the purge and trap device and analyze the same as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

11.7 Aqueous and Low level Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.7.1 Units which sample from the VOA vial should be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.7.2 If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. Note: Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.7.3 Soil samples must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1 g in 5 mL), if that preservation technique is used.

11.7.4 Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.7.5 For aqueous samples, check the pH of the sample remaining in the VOA vial after analysis is completed with narrow range pH paper. If the pH is greater than 2, a nonconformance memo should be initiated.

11.8 Low-Level Solids Analysis using discrete autosamplers

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846 and is not permitted within a number of programs including the OVAP and PADEP programs.

This method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogates, internal standards, and if applicable, the matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.8.1 Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.8.2 Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.01 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method described in section 11.7.

11.8.3 Connect the purge vessel to the purge and trap device.

11.8.4 Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from 11.8.2.

11.8.5 The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.8.6 Add the heater jacket or other heating device and start the purge and trap unit.

11.8.7 Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect.

11.9 Initial review and corrective actions

11.9.1 If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.9.2 If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.9.3 Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. Samples that are reported with internal standard exceedances must have documentation supporting matrix effect. Where the matrix effect is well established it may be reported with narration, otherwise the samples must be reanalyzed to confirm matrix effect is required. If the internal standard exceedance is deemed to be due to an instrumental problem, instrument maintenance will be done and all affected samples must be reanalyzed after the problem is corrected.

11.9.4 The surrogate standard recoveries are evaluated to ensure that they are within limits. See section 9.4 for corrective actions for surrogate recoveries.

11.10 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.10.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment.

11.10.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards, from the hardcopy printout of the “clean” reference spectrum book or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

12.1.1 The sample component retention time must compare to within at least ±0.06 RRT units of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

12.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.1.3 The relative intensities of ions should agree to within ±30% between the 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 percent.)

12.1.4 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.1.5 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.2 Tentatively Identified Compounds (TICs)

If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.2.2 The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

12.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.6 Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.3 Calculations.

12.3.1 Response factor (RF):

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

$\mathbf{A_x}$ = Area of the characteristic ion for the compound to be measured

$\mathbf{A_{is}}$ = Area of the characteristic ion for the specific internal standard

$\mathbf{C_{is}}$ = Concentration of the specific internal standard, ng

$\mathbf{C_x}$ = Concentration of the compound being measured, ng

Relative Retention Time (RRT) – is the ration of the retention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RT_C}{RT_{is}}$$

Where,

RT_C = Retention time for the volatile tragert compounds in the continuing calibration.

RT_{is} = Retention time for the internal standard in calibrtion standard or in a sample.

12.3.2 Standard deviation (SD):

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

$\mathbf{X_i}$ = Value of X at i through N

N = Number of points

X = Average value of $\mathbf{X_i}$

12.3.3 Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = **Mean of RF values in the curve**

12.3.4 Percent deviation between the initial calibration and the continuing calibration (%D):

Equation 4

$$\%\ \text{Deviation} = \frac{RRFic - RRFcc}{RRFic} \times 100$$

12.3.5 Percent drift between the initial calibration and the continuing calibration:

Equation 5

$$\% \text{ Drift} = \frac{C_{expected} - C_{found}}{C_{expected}} \times 100$$

Where

$C_{expected}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.6 Target compound and surrogate concentrations:

Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is $\leq$ 15%.

12.3.6.1 Calculation of concentration using Average Response Factors

Equation 6

$$\text{Concentration } \mu g / L = \frac{x}{RF}$$

12.3.6.2 Calculation of concentration using Linear fit

Equation 7

$$\text{Concentration } \mu g / L = A + Bx$$

12.3.6.3 Calculation of concentration using Quadratic fit

Equation 8

$$\text{Concentration } \mu g / L = A + Bx + Cx^2$$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.6.4 Calculation of **x** for Water and water-miscible waste:

Equation 9

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

$\mathbf{A_x}$ = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

$\mathbf{A_{is}}$ = Area of the characteristic ion for the internal standard

$\mathbf{I_s}$ = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

$\mathbf{V_o}$ = Volume of water purged, mL

12.3.6.5 Calculation of **x** for Medium level soils:

Equation 10

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

$\mathbf{A_x}$, $\mathbf{I_s}$, $\mathbf{D_f}$, $\mathbf{A_{is}}$, same as for water.

$\mathbf{V_t}$ = Volume of total extract, mL

$\mathbf{V_a}$ = Volume of extract added for purging, µL

$\mathbf{W_s}$ = Weight of sample extracted, g

$$\mathbf{D} = \frac{100 - \%\text{moisture}}{100}$$

12.3.6.6 Calculation of **x** for Low level soils:

Equation 11

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

$\mathbf{A_x}$, $\mathbf{I_s}$, $\mathbf{A_{is}}$, same as for water.

D is as for medium level soils

$\mathbf{W_s}$ = Weight of sample added to the purge vessel, g

12.3.6.7 Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

$\mathbf{A_x}$ = Area in the total ion chromatogram for the compound being measured

$\mathbf{A_{is}}$ = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to **x** as defined in equations 8, 9 and 10.

12.3.7 MS/MSD Recovery

Equation 12

$$\textbf{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result

SR = Sample result

SA = Spike added

12.3.8 Relative % Difference calculation for the MS/MSD

Equation 13

$$\textbf{RPD} = \frac{|\textbf{MSR - MSDR}|}{\frac{1}{2}(\textbf{MSR + MSDR})} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

13 METHOD PERFORMANCE

13.1 Method Detection Limit

Generally, each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA SOP # PITT-QA-0007. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method.

13.2 Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. The QC check sample is made up at 20 µg/L. (Some compounds will be at higher levels, refer to the calibration standard levels for guidance.)

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.2 The performance of all four QC check samples must meet all method requirements for LCSs.

13.2.3 If any analyte does not meet the acceptance criteria, check the acceptance limits in the reference methods (Table 6 of Method 8260B). If the recovery or precision is outside the limits in the reference methods, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14 POLLUTION PREVENTION

14.1 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 This method does not contain any specific modifications that serve to minimize or prevent pollution.

15 WASTE MANAGEMENT

15.1 The following waste streams are produced when this method is carried out.

15.1.1 Aqueous waste generated from analysis. This material may have a pH of less than 2.0. This waste is collected in containers identified as "Acid Waste", Waste #33. It is neutralized to a pH between 6 and 9 and disposed down a lab sink.

15.1.1 Solvent waste generated from analysis. This waste is placed in containers identified as "Vials & Extracts", Waste #7.

15.1.2 Solid waste generated from analysis. This waste is placed in trash containers and disposed with other building trash.

15.1.3 Expired Standards. This waste is placed in container identified as "Mixed Flammable Solvent Waste", Waste #3.

16 REFERENCES

16.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996.

16.2 40 CFR Chapter I Part 136, Appendix A, Method 624, 7-1-1997 Edition.

16.3 SOP # PITT-QA-DoD-0001, Implementation of DoD QSM Version 3 January 2006, current version.

16.4 USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, OSWER 9240.1-05A-P, PG99-963-506, EPA540/R-99/008, October 1999.

16.5 SOP # PITT-QA-0007, Determination of Method Detection Limits (MDL).

16.6 SOP # S-Q-004, Acceptable Manual Integration Practices.

17 MISCELLANEOUS

17.1 Modifications from SW-846 Method 8260B

17.1.1 Ion 119 is used as the quantitation ion for chlorobenzene-d5.

17.1.2 A relative retention time window of ±0.06 RT units is used for all components.

17.1.3 The quantitation and qualifier ions for some compounds have been added to the list of those which are recommended in SW-846 in order to improve the reliability of qualitative identification.

17.2 Modification from Method 5035

17.2.1 Presence of residual chlorine is not tested for water samples in section 8.2

17.2.2 Soils samples are not preserved with sodium bisulfate in section 8.4 for low level soils. Refer to sections 8.4 and 8.9.

17.2.3 Flow diagram for Field bisulfate preservation procedure was removed.

17.3 Other Modification in this version of SOP are highlighted.

17.4 Flow diagrams

Initial Demonstration and MDL

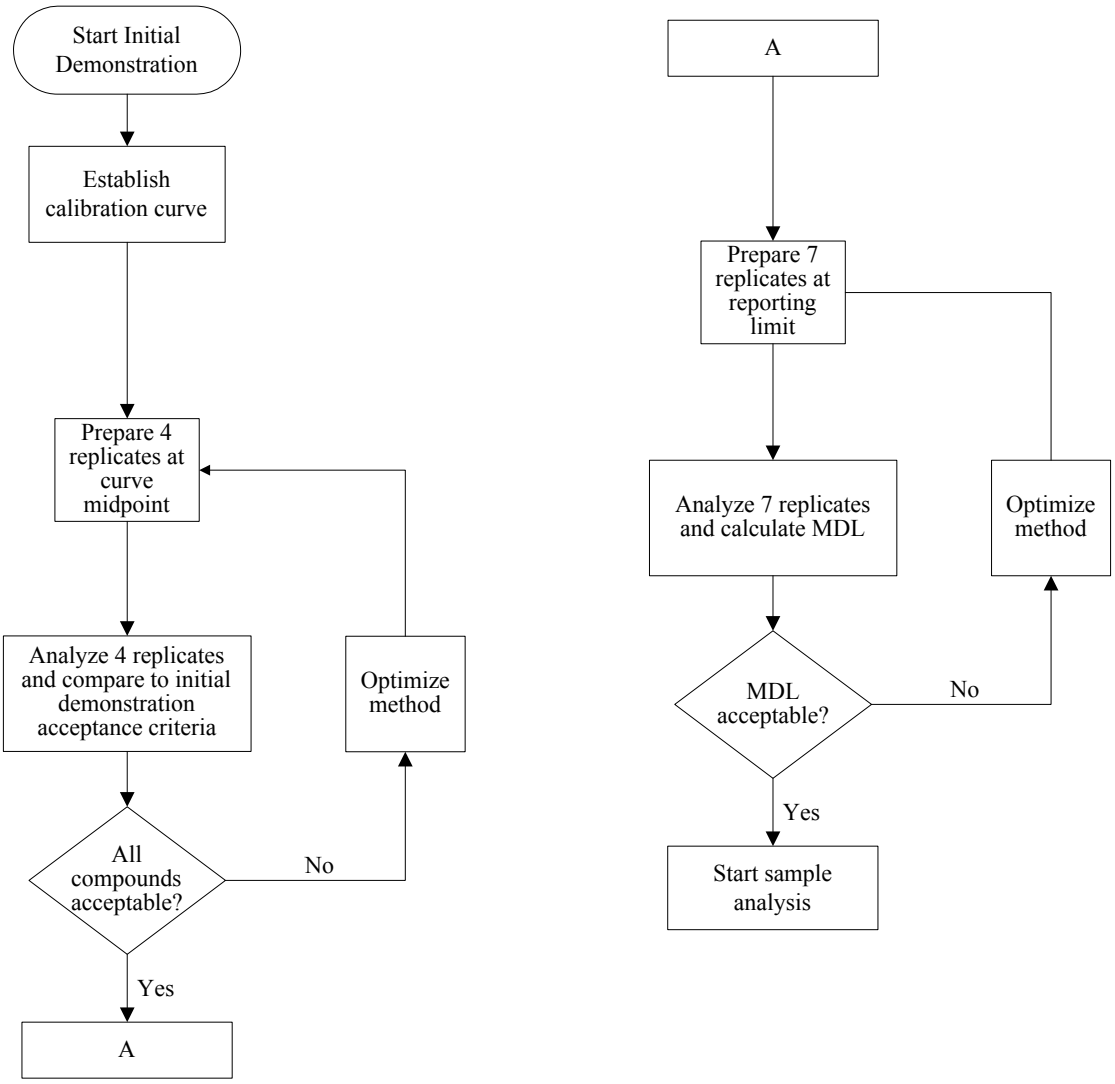

Start Initial Demonstration
Establish calibration curve
Prepare 4 replicates at curve midpoint
Analyze 4 replicates and compare to initial demonstration acceptance criteria
Optimize method
All compounds acceptable?
No
Yes
A
A
Prepare 7 replicates at reporting limit
Analyze 7 replicates and calculate MDL
Optimize method
MDL acceptable?
No
Yes
Start sample analysis

Table 1
TestAmerica Primary Standard and Reporting Limits for SW846 8260B

Compound	CAS Number	Low Level water µg/L	5 mL Water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	1	5	5	250
Chloromethane	74-87-3	1	5	5	250
Bromomethane	74-83-9	1	5	5	250
Vinyl chloride	75-01-4	1	5	5	250
Chloroethane	75-00-3	1	5	5	250
Trichlorofluoromethane	75-69-4	1	5	5	250
Acetone	67-64-1	10	20	20	1000
Trichlorotrifluoroethane	76-13-1	1	5	5	250
Carbon disulfide	75-15-0	1	5	5	250
Methylene chloride	75-09-2	1	5	5	250
1,1-Dichloroethene	75-35-4	1	5	5	250
1,1-Dichloroethane	75-34-3	1	5	5	250
trans-1,2-Dichloroethene	156-60-5	1	5	5	250
Methyl tert-butyl ether (MTBE)	1634-04-4	1	5	5	250
cis-1,2-Dichloroethene	156-59-2	1	5	5	250
1,2-Dichloroethene (Total)	540-59-0	1	5	5	250
Chloroform	67-66-3	1	5	5	250
1,2-Dichloroethane	107-06-2	1	5	5	250
Dibromomethane	74-95-3	1	5	5	250
2-Butanone	78-93-3	5	20	20	1000
1,1,1-Trichloroethane	71-55-6	1	5	5	250
Carbon tetrachloride	56-23-5	1	5	5	250
Bromodichloromethane	75-27-4	1	5	5	250
1,2-Dichloropropane	78-87-5	1	5	5	250
cis-1,3-Dichloropropene	10061-01-5	1	5	5	250
Trichloroethene	79-01-6	1	5	5	250
Dibromochloromethane	124-48-1	1	5	5	250
1,2-Dibromoethane	106-93-4	1	5	5	250
1,2,3-Trichloropropane	96-18-4	1	5	5	250
1,1,2-Trichloroethane	79-00-5	1	5	5	250
Benzene	71-43-2	1	5	5	250
trans-1,3-Dichloropropene	10061-02-6	1	5	5	250
Bromoform	75-25-2	1	5	5	250
4-Methyl-2-pentanone	108-10-1	5	20	20	1000
2-Hexanone	591-78-6	5	20	20	1000

Table 1
TestAmerica Primary Standard and Reporting Limits for SW846 8260B

Compound	CAS Number	Low Level water µg/L	5 mL Water µg/L	Low soil µg/kg	Med. Soil µg/kg
Tetrachloroethene	127-18-4	1	5	5	250
Toluene	108-88-3	1	5	5	250
1,1,2,2-Tetrachloroethane	79-34-5	1	5	5	250
1,1,1,2-Tetrachloroethane	630-20-6	1	5	5	250
1,2-Dibromo-3-chloropropane	96-12-8	1	5	5	250
Chlorobenzene	108-90-7	1	5	5	250
Ethylbenzene	100-41-4	1	5	5	250
Styrene	100-42-5	1	5	5	250
m and p Xylenes		2	10	10	500
o-xylene	95-47-6	1	5	5	250
Total xylenes	1330-20-7	3	15	15	750
p-Isopropyltoluene	99-87-6	1	5	5	250
Methylcyclohexane	108-87-2	1	5	5	250
1,1,2-Trichloro-1,2,2-Trifluoroethane	76-13-1	1	5	5	250
Methyl acetate	79-20-9	1	5	5	250
Cyclohexane	110-82-7	1	5	5	250
1,3-Dichlorobenzene	541-73-1	1	5	5	250
1,4-Dichlorobenzene	106-46-7	1	5	5	250
1,2-Dichlorobenzene	95-50-1	1	5	5	250
Isopropylbenzene	98-82-8	1	5	5	250
Bromobenzene	108-86-1	1	5	5	250
n-Propylbenzene	103-65-1	1	5	5	250
2-Chlorotoluene	95-49-8	1	5	5	250
4-Chlorotoluene	106-43-4	1	5	5	250
1,3,5-Trimethylbenzene	108-67-8	1	5	5	250
tert-Butylbenzene	98-06-6	1	5	5	250
1,2,4-Trimethylbenzene	95-63-6	1	5	5	250
sec-Butylbenzene	135-98-8	1	5	5	250
n-Butylbenzene	104-51-8	1	5	5	250
1,2,4-Trichlorobenzene	120-82-1	1	5	5	250
Naphthalene	91-20-3	1	5	5	250
Hexachlorobutadiene	87-68-3	1	5	5	250
1,2,3-Trichlorobenzene	87-61-6	1	5	5	250

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table 2
TestAmerica Appendix IX Standard and Reporting Limits for SW846 8260B

Compound	CAS Number	Low level water µg/L	5 mL Water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	1	5	5	250
Acetonitrile	75-05-8	20	100	100	5000
Acrolein	107-02-8	20	100	100	5000
Chloroprene	126-99-8	1	5	5	250
Iodomethane	74-88-4	1	5	5	250
Propionitrile	107-12-0	2	10	10	500
Methacrylonitrile	126-98-7	1	5	5	250
Isobutanol	78-83-1	40	200	200	10000
Iodomethane	74-88-4	1	5	5	250
Methyl methacrylate	80-62-6	1	5	5	250
Acrylonitrile	107-13-1	20	100	100	5000
Ethylmethacrylate	97-63-2	1	5	5	250
2-Chloroethyl vinyl ether[1]	110-75-8	2	10	10	500
tert-Butyl Alcohol	75-65-0	40	200	200	10,000
1,4-Dioxane	123-91-1	200	1000	1000	50000
Vinyl acetate	108-05-4	1	5	5	250
t-1,4-Dichloro-2-butene	110-57-6	1	5	5	250

Table 3

TestAmerica Primary Standard Calibration Levels, Standard 5 mL purge (Low Level Calibration Levels)

Compound	Calibration Level (ug/L)					
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
1,2-Dichloroethane-d4 (Surrogate)	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Toluene-d8 (Surrogate)	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
4-Bromofluorobenzene (Surrogate)	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Dichlorodifluoromethane *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Chloromethane *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Bromomethane *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Vinyl chloride *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Chloroethane *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Trichlorofluoromethane *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Acetone *	5 (2)	25 (10)	50 (20)	100 (30)	200 (40)	250 (80)
Carbon disulfide *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Methylene chloride	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Isopropylbenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,1-Dichloroethene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,1-Dichloroethane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
trans-1,2-Dichloroethene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,1,1,2-Tetrachloroethane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Methyl tert-butyl ether (MTBE) *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,2-Dibromo-3-chloropropane *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
cis-1,2-Dichloroethene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Chloroform	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,2-Dichloroethane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Dibromomethane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
2-Butanone *	5 (2)	25 (10)	50 (20)	100 (30)	200 (40)	250 (80)
1,1,1-Trichloroethane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Carbon tetrachloride	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Bromodichloromethane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,2-Dichloropropane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
cis-1,3-Dichloropropene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Trichloroethene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Dibromochloromethane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,2-Dibromoethane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,2,3-Trichloropropane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)

Table 3

TestAmerica Primary Standard Calibration Levels, Standard 5 mL purge (Low Level Calibration Levels)

Compound	Calibration Level (ug/L)					
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
1,1,2-Trichloroethane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Benzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
trans-1,3-Dichloropropene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Bromoform	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
4-Methyl-2-pentanone *	5 (2)	25 (10)	50 (20)	100 (30)	200 (40)	250 (80)
2-Hexanone *	5 (2)	25 (10)	50 (20)	100 (30)	200 (40)	250 (80)
Tetrachloroethene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Toluene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,1,2,2-Tetrachloroethane	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Chlorobenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Ethylbenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Styrene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
m and p Xylenes	10 (2)	50 (10)	100 (20)	200 (30)	400 (40)	500 (80)
o-xylene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,3-Dichlorobenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,4-Dichlorobenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,2-Dichlorobenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Isopropylbenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Bromobenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
n-Propylbenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
2-Chlorotoluene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
4-Chlorotoluene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,3,5-Trimethylbenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
tert-Butylbenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,2,4-Trimethylbenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
sec-Butylbenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
n-Butylbenzene	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,2,4-Trichlorobenzene *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Naphthalene *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Hexachlorobutadiene *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
1,2,3-Trichlorobenzene *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)

For medium level soils the above standard concentrations will be multiplied by 50.

Table 4

TestAmerica Appendix IX Standard Calibration Levels, Standard 5 mL purge (Low Level Calibration Levels)

Compound	Calibration Level (ug/L)					
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Allyl Chloride *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Acetonitrile *	100 (20)	500 (100)	1000 (200)	2000 (300)	4000 (400)	5000 (800)
Chloroprene *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Propionitrile *	10 (2)	50 (10)	100 (20)	200 (30)	400 (40)	500 (80)
Methacrylonitrile *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Isobutanol *	200 (40)	1000 (200)	2000 (400)	4000 (600)	8000 (800)	10000 (1600)
Methyl methacrylate *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Acrolein *	100 (20)	125 (25)	150 (30)	175 (35)	200 (40)	250 (50)
1,4-Dioxane *	1000 (200)	5000 (1000)	10000 (2000)	20000 (3000)	40000 (4000)	50000 (8000)
tert-Butyl alcohol *	200 (40)	1000 (200)	2000 (400)	4000 (600)	8000 (800)	10000 (1600)
Acrylonitrile *	100 (20)	125 (25)	150 (30)	175 (35)	200 (40)	250 (50)
Ethylmethacrylate *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
2-Chloroethyl vinyl ether*	10 (2)	50 (10)	100 (20)	200 (30)	400 (40)	500 (80)
Vinyl Acetate *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
t-1,4-Dichloro-2-butene*	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)
Iodomethane *	5 (1)	25 (5)	50 (10)	100 (15)	200 (20)	250 (40)

* Poor method performers (see section 10.4.11)

For medium level soils the above standard concentrations will be multiplied by 50.

Table 4A – Calibration Standard Concentration and Preparation

Standard Level 8260B Water or Soil 5mL syringe

STD	INT (25□g/mL)	SURR (25□g/mL)	VOA (25□g/mL)	Acetonitrile (1000□g/mL)	Methanol Added
5ppb	10ml	1ul	1ul	0.5ul	122.5ul
25ppb	10ul	5ul	5ul	2.5ul	112.5ul
50ppb	10ul	10ul	10ul	5ul	100ul
100ppb	10ul	20ul	20ul	10ul	75ul
200ppb	10ul	40ul	40ul	20ul	25ul
250ppb	10ul	50ul	50ul	25ul	0

8260B App IX Water/Soil 5mL syringe

STD	INT 25□g/mL	App IX 25□g/mL	2CEVE 50□g/mL	A&A 25□g/mL	TBA 1000□g/mL	n-Heptane 25□g/mL
5ppb	10ul	1ul	1ul	20ul	1ul	1ul
25ppb	10ul	5ul	5ul	25ul	5ul	5ul
50ppb	10ul	10ul	10ul	30ul	10ul	10ul
100ppb	10ul	20ul	20ul	35ul	20ul	20ul
200ppb	10ul	40ul	40ul	40ul	40ul	40ul
250ppb	10ul	50ul	50ul	50ul	50ul	50ul

Table 4A – Calibration Standard Concentration and Preparation Cont.

Dupont 5mL syringe

STD	INT 25□g/mL	Dupont VOA 25□g/mL	Dupont Acrylates 25□g/mL
5ppb	10ul	1ul	1ul
25ppb	10ul	5ul	5ul
50ppb	10ul	10ul	10ul
100ppb	10ul	20ul	20ul
200ppb	10ul	40ul	40ul
250ppb	10ul	50ul	50ul

CLP OLM04.1/3.1/3.2 Water & Soil 5mL syringe

STD	INT (25□g/mL)	SURR (25□g/mL)	VOA (25□g/mL)	Methanol Added
10ppb	10ul	2ul	2ul	80ul
20ppb	10ul	4ul	4ul	70ul
50ppb	10ul	10ul	10ul	60ul
100ppb	10ul	20ul	20ul	40ul
200ppb	10ul	40ul	40ul	0

Table 4A – Calibration Standard Concentration & Preparation Cont.

624 & Low Level 8260B Water 25mL syringe

STD	INT 25□g/mL	SURR (25□g/mL)	VOA (25□g/mL)	Acetonitrile (1000□g/mL)	Ketone 25□g/mL	Methanol Added
1ppb	10ul	1ul	1ul	0.5ul	4ul	140ul
5ppb	10ul	5ul	5ul	2.5ul	5ul	120ul
10ppb	10ul	10ul	10ul	5.0ul	10ul	105ul
15ppb	10ul	15ul	15ul	7.5ul	15ul	88ul
20ppb	10ul	20ul	20ul	10ul	20ul	70ul
40ppb	10ul	40ul	40ul	20ul	40ul	0

8260B Low Level App IX Water 25mL syringe

STD	INT 25□g/mL	App IX (Various)	2CEVE 50□g/mL	A&A 25□g/mL	TBA 1000□g/mL
5ppb	10ul	1ul	1ul	20ul	1ul
25ppb	10ul	5ul	5ul	25ul	5ul
50ppb	10ul	10ul	10ul	30ul	10ul
75ppb	10ul	15ul	15ul	35ul	15ul
100ppb	10ul	20ul	20ul	40ul	20ul
200ppb	10ul	40ul	40ul	50ul	40ul

Table 4-1
8260 ICal Control List
12/05/2003

Compound	SW-846	Control	SPCC
1,1-Dichloroethene	8260B	CCC	
Chloroform	8260B	CCC	
Ethylbenzene	8260B	CCC	
Toluene	8260B	CCC	
Vinyl Chloride	8260B	CCC	
1,2-Dichloropropane	8260B	CCC	
1,1,1-Trichloroethane	8260B	1	
1,1,2,2-Tetrachloroethane	8260B	1	
1,1,2-Trichloroethane	8260B	1	SPCC
1,1-Dichloroethane	8260B	1	
1,2-Dichlorobenzene	8260B	1	SPCC
1,2-Dichloroethane	8260B	1	
1,3-Dichlorobenzene	8260B	1	
1,4-Dichlorobenzene	8260B	1	
Benzene	8260B	1	
Bromodichloromethane	8260B	1	
Bromoform	8260B	1	SPCC
Bromomethane	8260B	1	
Carbon Tetrachloride	8260B	1	
Chlorobenzene	8260B	1	SPCC
Cis-1,3-Dichloropropene	8260B	1	
Dibromochloromethane	8260B	1	
Styrene	8260B	1	
Tetrachloroethene	8260B	1	
Trans-1,3-Dichloropropene	8260B	1	
Trichloroethene	8260B	1	
Xylenes (total)	8260B	1	
1,2,4-Trichlorobenzene	8260B	1	
1,1,2-Trochloro-1,2,2-Trifluoroethane	8260B	2	
1,2-Dibromo-3-Chloropropane	8260B	2	
1,2-Dibroethane	8260B	2	
1,2-Dichloroethene (total)	8260B	2	
2-Butanone	8260B	2	

Table 4-1
8260 ICal Control List
12/05/2003

Compound	SW-846	Control	SPCC
2-Hexanone	8260B	2	
4-Methyl-2-Pentanone	8260B	2	
Acetone	8260B	2	
Carbon Disulfide	8260B	2	
Chloroethane	8260B	2	
Chloromethane	8260B	2	SPCC
Cis-1,2-Dichloroethene	8260B	2	
Cyclohexane	8260B	2	
Dichlorodifluoromethane	8260B	2	
Isopropylbenzene	8260B	2	
Methyl Acetate	8260B	2	
Methyl Tert-Butyl Ether	8260B	2	
Methylcyclohexane	8260B	2	
Methylene Chloride	8260B	2	
Trans-1,2-Dichloroethene	8260B	2	
Trichlorofluoromethane	8260B	2	
1,1,1,2-Tetrachloroethane	8260B	2	
1,2-Dichloropropene	8260B	2	
1,2,3-Trichlorobenzene	8260B	2	
1,2,3-Trichloropropane	8260B	2	
1,2,4-Trimethylbenzene	8260B	2	
1,2-Dibromoethane (EDB)	8260B	2	
1,3,5-Trimethylbenzene	8260B	2	
1,3-Dichloropropane	8260B	2	
1,4-Dioxane	8260B	2	
2,2-Dichloropropane	8260B	2	
2-Butanone (MEK)	8260B	2	
2-Chloroethyl Vinyl Ether	8260B	2	
2-Chlorotoluene	8260B	2	
4-Chlorotoluene	8260B	2	
4-Methyl-2-Pentanone (MIBK)	8260B	2	
Acetonitrile	8260B	2	
Acrolein	8260B	2	
Acrylonitrile	8260B	2	

Table 4-1
8260 ICal Control List
12/05/2003

Compound	SW-846	Control	SPCC
Ally Chloride	8260B	2	
Bromobenzene	8260B	2	
Bromochloromethane	8260B	2	
Chlorodibromomethane	8260B	2	
Chloroprene	8260B	2	
Dibromomethane	8260B	2	
Dichlorobromemethane	8260B	2	
Ethyl Methacrylate	8260B	2	
Hexachlorobutadiene	8260B	2	
Iodomethane	8260B	2	
Isobutanol	8260B	2	
Isobutyl Alcohol	8260B	2	
m-Dichlorobenzene	8260B	2	
Methacrylonitrile	8260B	2	
Methyl Bromide	8260B	2	
Methyl Chloride	8260B	2	
Methyl Ethyl Ketone	8260B	2	
Methyl Iodide	8260B	2	
Methyl Methacrylate	8260B	2	
Methyl Tert-Butyl Ether (MTBE)	8260B	2	
Methylene Bromide	8260B	2	
m-Xylene & p-Xylene	8260B	2	
Naphthalene	8260B	2	
n-Butylbenzene	8260B	2	
n-Propylbenzene	8260B	2	
o-Dichlorobenzene	8260B	2	
o-Xylene	8260B	2	
p-Dichlorobenzene	8260B	2	
p-Isopropyltoluene	8260B	2	
Propionitrile	8260B	2	
Sec-Butylbenzene	8260B	2	
Tert-Butylbenzene	8260B	2	
Tetrachloroethene	8260B	2	
Trnas-1,4-Dichloro-2-butene	8260B	2	

Table 4-1
8260 ICal Control List
12/05/2003

Compound	SW-846	Control	SPCC
Vinyl Acetate	8260B	2	

Narrative Issues:

- All %RSD that >30% must be narrated. This may be changed with the development of a calibration summary sheet.
- All %Diff or %Drift >25% must be narrated.
- Any other criteria exceedance aside from these should be narrated.
- Using an average response factor curve for a %RDS ≥30% should be narrated.
- If a list two compound > 50% D or Drift and is out high and this compound is not found in the associated samples it may be reported with narration.

  Note: These criterion are subject to project-specific criteria which may vary depending on project needs.

Table 5
Reportable Analytes for TestAmerica Standard Tests

Compound	CAS Number	624	8260	Appendix IX	CLP 4.2
Dichlorodifluoromethane	75-71-8		X	X	X
Chloromethane	74-87-3	X	X	X	X
Bromomethane	74-83-9	X	X	X	X
Vinyl chloride	75-01-4	X	X	X	X
Chloroethane	75-00-3	X	X	X	X
Trichlorofluoromethane	75-69-4	X	X	X	X
Acrolein	107-02-8			X	
Acetone	67-64-1		X	X	X
Iodomethane	74-88-4			X	
Carbon disulfide	75-15-0		X	X	X
Methylene chloride	75-09-2	X	X	X	X
tert-Butyl alcohol	75-65-0			X	
1,1-Dichloroethene	75-35-4	X	X	X	X
1,1-Dichloroethane	75-34-3	X	X	X	X
trans-1,2-Dichloroethene	156-60-5	X	X	X	X
Acrylonitrile	107-13-1			X	
Methyl tert-butyl ether (MTBE)	1634-04-4	X	X	X	X
cis-1,2-Dichloroethene	156-59-2		X	X	X
Chloroform	67-66-3	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X
Dibromomethane	74-95-3		X	X	
2-Butanone	78-93-3		X	X	X
1,4-Dioxane	123-91-1			X	
1,1,1-Trichloroethane	71-55-6	X	X	X	X
Carbon tetrachloride	56-23-5	X	X	X	X
Bromodichloromethane	75-27-4	X	X	X	X
1,2-Dichloropropane	78-87-5	X	X	X	X
cis-1,3-Dichloropropene	10061-01-5	X	X	X	X
Trichloroethene	79-01-6	X	X	X	X
Dibromochloromethane	124-48-1	X	X	X	X
1,2-Dibromoethane	106-93-4		X	X	X
1,2,3-Trichloropropane	96-18-4		X	X	
1,1,2-Trichloroethane	79-00-5	X	X	X	X
Benzene	71-43-2	X	X	X	X

Table 5
Reportable Analytes for TestAmerica Standard Tests

Compound	CAS Number	624	8260	Appendix IX	CLP 4.2
Ethylmethacrylate	97-63-2			X	
trans-1,3-Dichloropropene	10061-02-6	X	X	X	X
Bromoform	75-25-2	X	X	X	X
4-Methyl-2-pentanone	108-10-1		X	X	X
2-Hexanone	591-78-6		X	X	X
Tetrachloroethene	127-18-4	X	X	X	X
Toluene	108-88-3	X	X	X	X
1,1,2,2-Tetrachloroethane	79-34-5	X	X	X	X
2-Chloroethyl vinyl ether	110-75-8	X		X	
Vinyl acetate	108-05-4			X	
Chlorobenzene	108-90-7	X	X	X	X
Ethylbenzene	100-41-4	X	X	X	X
Styrene	100-42-5		X	X	X
t-1,4-Dichloro-2-butene	110-57-6			X	
m and p Xylenes			X	X	
o-xylene	95-47-6		X	X	
Total xylenes	1330-20-7		X	X	X
1,3-Dichlorobenzene	541-73-1	X	X		X
1,4-Dichlorobenzene	106-46-7	X	X		X
1,2-Dichlorobenzene	95-50-1	X	X		X
1,2-Dichloroethene (total)	540-59-0		X		
2,2-Dichloropropane	590-20-7		X	X	
Bromochloromethane	74-97-5		X		
1,1-Dichloropropene	563-58-6		X		
1,3-Dichloropropane	142-28-9		X		
1,1,1,2-Tetrachloroethane	630-20-6		X	X	
Isopropylbenzene	98-82-8		X		X
Bromobenzene	108-86-1		X		
n-Propylbenzene	103-65-1		X		
2-Chlorotoluene	95-49-8		X		
4-Chlorotoluene	106-43-4		X		
1,3,5-Trimethylbenzene	108-67-8		X		
tert-Butylbenzene	98-06-6		X		
1,2,4-Trimethylbenzene	95-63-6		X		

Table 5
Reportable Analytes for TestAmerica Standard Tests

Compound	CAS Number	624	8260	Appendix IX	CLP 4.2
sec-butylbenzene	135-98-8		X		
4-Isopropyltoluene	99-87-6		X		
n-Butylbenzene	104-51-8		X		
1,2-Dibromo-3-chloropropane	96-12-8		X		X
1,2,4-Trichlorobenzene	120-82-1		X		X
Napthalene	91-20-3		X		
Hexachlorobutadiene	87-68-3		X		
1,2,3-Trichlorobenzene	87-61-6		X		
Methylcyclohexane	108-87-2		X		X
1,1,2-Trichloro-1,2,2-Triflroroethane	76-13-1		X		X
Methyl Acetate	79-20-9		X		X
Allyl Chloride	107-05-1			X	
Acetonitrile	75-05-8			X	
Chloroprene	126-99-8			X	
Propionitrile	107-12-0			X	
Methacrylonitrile	126-98-7			X	
Isobutanol	78-83-1			X	
Methyl methacrylate	80-62-6			X	

Table 6
Internal Standards

Internal Standard Compound	Standard Concentration µg/mL	Quantitation ion (m/z)
Fluorobenzene	25	96
Chlorobenzene-d5	25	119
1,4-Dichlorobenzene-d4	25	152

Notes:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a standard 5 mL purge Method 8260B, or 10 µg/L for low level Method 8260B waters (which uses a 25 ml sample aliquot), Method 624. For instruments that sample directly from the VOA vial, 10 µL of a 5 µg/mL internal standard solution is added to low level Method 8260B waters, and Method 624 since the instrument uses a 5 ml sample volume.
2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 7
Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	25
Dibromofluoromethane	25
Toluene-d_8	25
4-Bromofluorobenzene	25

Notes:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a standard 5 mL purge Method 8260B, or 10 µg/L for low level Method 8260B waters (which uses a 25 ml sample aliquot), Method 624. For instruments that sample directly from the VOA vial, 10 µL of a 5 µg/mL surrogate solution is added to low level Method 8260B waters, and Method 624 since the instrument uses a 5 ml sample volume.
2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.
3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 8
Matrix Spike / LCS Compounds

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	25
Trichloroethene	25
Toluene	25
Benzene	25
Chlorobenzene	25

Notes:

1) 10 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a standard 5 mL purge Method 8260B water or 10 µg/L for a low level Method 8260B sample when added to a 25 ml sample aliquot.
2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by the QA department.

Table 9
BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 10
SPCC Compounds and Minimum Response Factors

Compound	8260B Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	>0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 11
CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	≤30.0	≤20.0
1,1-Dichloroethene	≤30.0	≤20.0
Chloroform	≤30.0	≤20.0
1,2-Dichloropropane	≤30.0	≤20.0
Toluene	≤30.0	≤20.0
Ethylbenzene	≤30.0	≤20.0

Table 12
Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Cyclohexane	56	69	84
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl tert butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98

Table 12
Characteristic ions

Compound	Primary*	Secondary	Tertiary
2-Butanone	43	72**	
Tetrahydrofuran (THF)	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78

Table 12
Characteristic ions

Compound	Primary*	Secondary	Tertiary
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.
** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

Table 13 - 8260B QC Acceptance Criteria														
	Water	LCS			MS			Soil	LCS			MS		
Compound	AMT ug/L	LCL	UCL	RPD	LCL	UCL	RPD	AMT ug/kg	LCL	UCL	RPD	LCL	UCL	RPD
Acetone	50	11	129	32	10	129	50	50	10	186	79	10	186	79
Benzene	50	77	120	20	75	120	22	50	74	120	27	64	132	27
Bromodichloromethane	50	70	123	20	70	123	24	50	69	120	29	69	120	29
Bromoform	50	60	127	20	50	132	35	50	54	129	37	44	132	37
Bromomethane	50	51	154	23	51	154	44	50	30	154	54	30	154	54
2-Butanone	50	35	126	35	35	126	35	50	25	154	63	25	154	63
Carbon disulfide	50	64	126	20	59	130	20	50	57	127	35	57	127	35
Carbon tetrachloride	50	71	126	25	71	126	25	50	68	125	35	67	143	35
Chlorobenzene	50	78	120	20	78	120	23	50	77	120	25	61	133	25
Dibromochloromethane	50	69	123	20	67	123	29	50	67	121	30	66	123	30
Chloroethane	50	43	183	24	43	183	52	50	28	172	55	28	172	55
Chloroform	50	76	120	25	76	120	25	50	75	120	36	75	120	36
Chloromethane	50	56	124	25	56	124	27	50	36	140	50	36	140	50
Cyclohexane	50	50	150	50	50	150	50	50	50	150	50	50	150	50
1,2-Dibromo-3-chloropropane	50	41	131	20	41	145	20	50	29	144	72	10	150	72
1,2-Dibromoethane	50	67	120	20	67	120	20	50	62	122	20	57	127	20
1,2-Dichlorobenzene	50	67	120	20	67	120	20	50	72	120	30	72	120	30
1,3-Dichlorobenzene	50	72	120	21	72	120	21	50	75	120	30	75	120	30
1,4-Dichlorobenzene	50	74	120	20	74	120	20	50	75	120	30	75	120	30
Dichlorodifluoromethane	50	29	141	20	29	144	32	50	10	153	80	10	153	80
1,1-Dichloroethane	50	75	120	22	75	120	22	50	71	120	47	71	120	47
1,2-Dichloroethane	50	68	122	25	68	122	25	50	66	122	43	66	122	43
cis-1,2-Dichloroethene	50	75	120	20	75	120	23	50	72	120	20	72	120	20
trans-1,2-Dichloroethene	50	74	120	20	74	120	22	50	67	121	20	67	121	20
1,1-Dichloroethene	50	65	125	20	61	128	20	50	63	126	33	61	138	33
1,2-Dichloropropane	50	73	120	20	73	120	20	50	73	120	20	73	120	20
cis-1,3-Dichloropropene	50	73	120	20	73	120	25	50	71	120	40	71	120	40
trans-1,3-Dichloropropene	50	69	124	32	69	124	32	50	67	121	31	67	121	31

Table 13 - 8260B QC Acceptance Criteria														
	Water	LCS			MS			Soil	LCS			MS		
Compound	AMT ug/L	LCL	UCL	RPD	LCL	UCL	RPD	AMT ug/kg	LCL	UCL	RPD	LCL	UCL	RPD
Ethylbenzene	50	77	120	25	77	120	25	50	77	120	25	76	128	25
2-Hexanone	50	34	130	24	34	130	24	50	33	147	31	33	147	31
Isopropylbenzene	50	72	124	20	71	124	25	50	71	125	20	71	125	20
Methyl acetate	50	10	150	50	10	150	50	50	10	150	50	10	150	50
Methylcyclohexane	50	50	150	50	50	150	50	50	50	150	50	50	150	50
Methylene chloride	50	71	120	20	71	120	22	50	66	129	20	65	134	20
4-Methyl-2-pentanone	50	50	136	34	50	136	34	50	42	139	39	37	146	39
Methyl tert-butyl ether	50	60	126	50	56	128	50	50	55	126	45	47	131	45
Styrene	50	75	120	22	75	120	25	50	73	121	22	73	121	22
1,1,2,2-Tetrachloroethane	50	60	123	20	60	123	20	50	51	130	20	38	138	20
Tetrachloroethene	50	70	122	25	70	122	25	50	73	120	25	73	120	25
Toluene	50	76	120	20	76	120	23	50	75	120	26	60	134	26
1,2,4-Trichlorobenzene	50	33	149	30	33	146	30	50	48	131	30	48	131	30
1,1,1-Trichloroethane	50	74	121	24	74	121	24	50	71	121	24	71	121	24
1,1,2-Trichloroethane	50	68	120	23	68	120	23	50	64	121	23	61	125	23
Trichloroethene	50	77	120	20	77	120	23	50	73	120	26	52	143	26
Trichlorofluoromethane	50	17	174	20	17	174	50	50	21	153	20	21	153	20
1,1,2-Trichloro-1,2,2-trifluoroethane	50	62	122	30	53	131	30	50	54	129	30	53	146	30
Vinyl chloride	50	57	127	25	57	127	26	50	43	138	25	43	138	25
Xylenes (total)	150	76	120	20	76	120	24	150	75	121	20	75	121	20
4-Bromofluorobenzene	50	75	120		75	120		50	63	120		63	120	
1,2-Dichloroethane-d4	50	70	125		70	125		50	52	124		52	124	
Toluene-d8	50	80	120		80	120		50	72	127		72	127	
Dibromofluoromethane	50	80	120		80	120		50	68	121		68	121	

These limits are established based on internal laboratory data.

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Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
Breakdown Check 8081A: Endrin, DDT 8270C: DDT	Analysis of s standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degredation of either DDT or Endrin exceeds method–specified criteria, corrective action must be taken before proceeding with calibration.
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified.	To verify the identification of an analyte.	This is a required QC procedure. All positive results must be confirmed.
CCV	This verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.	To verify that instrument response is reliable, and has not changed significantly from the current ICAL.	If the values for the analytes are outside the acceptance criteria, the initial calibration may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging.
Demonstrate Acceptable Analyst Capability	Analyst runs QC samples in series to establish his/her ability to produce data of acceptable accuracy and precision.	To establish the analysts' ability to produce data of acceptable accuracy and precision.	The average recovery and standard deviation of the replicate must be within designated acceptance criteria.
Duplicate Sample	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are taken and prepared and analyzed identically.	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix.	To provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the sample and the duplicate.
ICAL	Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method.	To establish a calibration curve for the quantification of the analytes of interest.	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The ICAL must be successful before any samples or other QC check samples can be analyzed.
Internal Standards	A known amount of standard added to all standards and samples as a	To verify that the analytical system is in control.	Any sample associated with out-of-control results must be

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
	reference for evaluating and controlling the precision and bias of the applied analytical method.		reanalyzed.
LCS containing all analytes required to be reported	A QC standard of known composition prepared using reagent free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern.	To evaluate method performance by assessing the ability of the lab/analyst to successfully recover the target analytes from a control (clean) matrix.	This is a required QC Check. The inability to achieve acceptable recoveries in the LCS indicate problems with the accuracy/bias of the measurement system.
MS	A sample prepared by adding a know amount of targeted analyte(s) to an aliquot of a specific environmental sample.	To assess the performance of the method as applied to a particular matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The lab should not correct for recovery; only report the results of the analyses and the associated MS results and indicate that the results from these analyses have increased uncertainty.
MSD	A 2^{nd} replicate MS prepared in the lab, spiked with an identical, known amount of targeted analyte(s), and analyzed to obtain a measure of the precision of the recovery for each analyte.	To assess the performance of the method as applied to a particular matrix and provide information of the homogeneity of the matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix.
MDL Verification Check	A low-level spike taken through the prep and analytical steps at approximately 2x the MDL used to verify that the laboratory can detect analytes at the calculated MDL.	To validate the MDL on an ongoing basis	If the MDL verification check fails, reprep/reanalyze at a higher level to set a higher MDL or the MDL study must be repeated.
MB	A sample of a matrix similar to the batch of associated samples in which no target analytes or interferences are present at concentrations that impact the analytical results.	To assess background interferences or contamination in the analytical system that might lead to high bias or false positive data.	This QC is used to measure lab accuracy/bias. The MB could indicate whether contamination is occurring during sample prep and analysis. If analytes are detected > ½ RL, reanalyze or B-Flag

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
			results for all samples in prep batch. For common lab contaminants, no analytes detected > RL. See DoD Box D-5; & Sec. D.1.1.1
MDL Study	The process to determine the minimum concentration of an analyte 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.	To determine the lowest concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.	MDLs must be established prior to sample analysis. The RL or LOQ is at least 3x the MDL. Used in combination with the MDL verification check to validate the MDL on an ongoing basis.
RT window position establishment for each analyte (chromatographic methods only)	Determination of the placement of the RT window (start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the initial calibration.	To identify analytes of interest	Incorrect window position may result in false negatives, require additional manual integrations, and/or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
RT window verification for each analyte (chromatographic methods only)	A standard is used to verify that the width and position of the RT windows are valid so that accurate analyte identification can be made during sample analysis.	To minimize the occurrence of both false positive and false negative results at each calibration verification.	The peaks from the standard used are compared to the RT window established during the ICAL to verify that the analytes of interest still fall within the window.
Second source calibration verification	A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration.	To verify the accuracy of the initial calibration.	The concentration of the 2nd source calibration verification, determined from the analysis, is compared to the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.
Surrogate spike (organic analysis only)	A pure substance with properties that mimic the analyte of interest. Surrogates are compounds unlikely to be found in environmental samples to evaluate analytical efficiency by	To assess the ability of the method to successfully recover specific non-target analytes from an actual matrix.	Whereas the MS is normally done on a batch-specific basis, the surrogate spike is done on a sample-specific basis. Taken with the information derived from

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-1 Summary of QC Check Definitions, Purpose, and Evaluation – Organics (GC/MS)

QC Check	Definition	Purpose	Evaluation
	measuring their % Recovery.	matrix.	other spikes (LCS; MS), the bias in the analytical system can be determined.
Tuning (MS methods only)	The analysis of a standard compound to verify the mass spectrometer meets standard mass spectra abundance criteria prior to sample analysis.	To verify the proper working of the mass spectrometer.	Proper tuning of the mass spectrometer must be verified prior to sample analysis .

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.

2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

3. If the requirements in the DoD tables do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3: Organic Analysis by GC/MS - Methods 8260 and 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
IDOC	Per Instrument/Analyst	DoD acceptance criteria if available; otherwise method specific criteria.	Correct / Repeat for those analytes which failed criteria.	NA
MDL	Annually or quarterly MDL Checks performed	40 CFR 136B; MDL verification checks must produce a signal at least 3x the instrument's noise level.	Run MDL check at higher level and set MDL higher or reconduct MDL study.	NA
Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method specific ion criteria.	Retune instrument and verify. Rerun affected samples.	NA
Breakdown check DDT (8270C only)	Daily prior to analysis of samples	Degradation $\leq$ 20% for DDT (Benzidine & PCP should be present at their normal response and no peak tailing should be observed).	Correct problem then repeat breakdown check.	NA
ICAL	Initial 5-point calibration prior to sample analysis	1. Average RF for SPCCs: VOCs - $\geq$ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, $\geq$ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs - $\geq$ 0.050 2. RSD for RFs for CCCs: VOCs and SVOCs - $\leq$ 30% and one option below. **Option 1:** RSD for each analyte $\leq$ 15% **Option 2:** linear least squares regression: r $\geq$ 0.995 **Option 3:** non-linear regression: Coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for 2nd order, 7 points shall be	Correct problem then repeat initial calibration.	NA

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3: Organic Analysis by GC/MS - Methods 8260 and 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
		used for 3rd order)		
2nd Source calibration verification	Once after each initial calibration	Value of 2nd source for all analytes within ± 25% of expected value - See SOP Section 10.5.15 for exception an DoD SOP.	Correct problem and verify 2nd source standard. Rerun, if that fails, correct problem and repeat ICAL.	NA
RT window position establishement for each analyte	Once per ICAL	Position shall be set using midpoint standard of the initial calibration curve.	NA	NA
Evaluation of Relative RT (RRT)	With each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct problem, then rerun ICAL	NA
Calibration verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time	1. Average RF for SPCCs: VOCs - ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane SVOCs - ≥ 0.050 2. %Difference for CCCs: VOCs and SVOCs - ≤ 20% D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration) All calibration analytes must be within 20% D, with no individual analytes (except CCC's) > 25% D (DoD Version 2.2)	Correct problem, rerun CV, if fails, repeat ICAL (Data associated with an unacceptable CCV may be fully usable under the following conditions: 1. CCV (high bias) and samples ND, then raw data may be reported with appropriate flag 2. CCV (low bias) and samples exceed maximum regulatory limit/decision level (DoD Box 60: Project specific permission from appropriate DoD personnel is required to report data generated from a run with noncompliant CCV.)	Apply J-flag to all results associated with the analytical batch for all analytes > 20%D and < 25% D. Identify in case narrative analytes > 20% D. (DoD Version 2.2) Apply Q-flag if no sample material remains and analyte exceeds criteria
Internal Standards verification	In all field samples and standards	RT ± 30 seconds from RT of the midpoint standard in the ICAL EICP area within -50% to	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was	Apply Q-flag to analytes associated with the non-compliant IS.

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3: Organic Analysis by GC/MS - Methods 8260 and 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
		+100% of ICAL midpoint standard.	malfunctioning is mandatory.	compliant IS.
MB	One per prep batch	No analytes detected > ½ RL For common lab contaminants, no analytes > RL	Correct problem, then see criteria in box D-5; if required, reprep/reanalyze MB and all associated samples.	Apply B-flag to all results for the contaminated analyte for all samples in the associated prep batch.
LCS (containing all analytes to be reported)	One LCS per prep batch	DoD specified QC criteria, if available	Correct problem, reprep/reanalyze the LCS and all samples in the associated prep batch for all failed analytes, if sufficient sample is available.	Apply Q-flag to specific analyte(s) in all samples in the prep batch.
MS	One per prep batch per matrix	For matrix evaluation, use DoD specified QC criteria for LCS.	Examine the project-specific DQOs. Contact client for additional corrective action measures.	Apply J-flag to specific analyte(s) in the parent sample.
MSD or Sample Duplicate	One per prep batch per matrix	RPD $\leq$ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact client for additional corrective action measures.	Apply J-flag to specific analyte(s) in the parent sample.
Surrogate	All field and QC samples	DoD specified QC criteria if available, otherwise method specific criteria or lab's own in-house criteria.	For QC and field samples, correct problem, reprep/reanalyze all failed samples in the associated prep batch if sufficient sample material is available.	Apply J-flag for specific analyte(s) in all field samples collected from the same site matrix as the parent. Apply Q-flag to QC samples for specific analyte(s)
Results reported between LOD and LOQ			Apply J-flag to all results between LOD (MDL) and LOQ (RL)	
Manual Integration	When manual integrations are performed	Raw data shall include a complete audit trail for those manipulations, raw data output showing the results of the MI (i.e., chromatograms of manually integrated peaks), and notation of		Apply M-flag to MI data

DoD QSM Version 3: Appendix DOD-B Quality Control Requirements Summary

Table B-3: Organic Analysis by GC/MS - Methods 8260 and 8270

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
		rationale, date, and signature/initials of person performing manual operation.		

Notes:

1. Project-specific requirements identified by the client supersede any requirements listed. The requirements are meant to be default, to be used when project-specific direction based on DQOs is not available.

2. If there is a contradiction between the method and the DoD tables, the requirements specified in the tables shall be followed.

3. If the requirements in the DoD tables do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

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18 REQUIREMENTS FOR EPA 624

18.1 Method 624 is required for demonstration of compliance with NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table B-1.

18.1.1 The tune period for this method is defined as 24 hours after passing a 25 ug/ml BFB.

18.1.2 The initial calibration curve for this method requires at least three points.

18.2 Sample concentrations are calculated using the average RRF from the initial calibration curve.

18.2.1 Each target analyte is assigned to the closest eluting internal standard.

18.2.2 Initial demonstration of Proficiency

18.2.3 The spiking level for the four replicate initial demonstration of proficiency is 20 µg/L. The acceptance criteria are listed in Table B-2

18.3 Initial calibration curve requirements:

18.3.1 Target compounds listed in Method 624 must have RSD ≤ 35%.

18.3.2 If this requirement cannot be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.

18.3.3 For compounds not listed in Method 624, the average response factor will be used for quantitation.

18.3.4 The initial calibration is verified daily by the analysis of a 20 ug/L second source QC Check Standard.

18.4 Continuing calibration verification requirements:

18.4.1 The continuing calibration standard is the daily QC Check Standard. The acceptance criteria are listed in Table B-2.

18.5 LCS and MS/MSD requirements

18.5.1 The daily 20 ug/L QC Check Standard also serves as the LCS.

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18.5.2 The MS and MSD will be 20 ug/L for all compounds.

18.5.3 The recovery limits for MS/MSD and LCS recovery are listed in Table B-2.

18.5.4 The LCS and MS are required for 5% of the samples.

18.6 Method clarifications, modifications and additions

18.6.1 Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely employs the Supelco VOCARB 3000, which consists of Carbopack B and Carboxen 1000 and 1001.

18.6.2 Section 5.3.2 of the source method describes a packed analytical column. TestAmerica routinely employs capillary columns when performing this method.

18.6.3 The source method provides a suggested list of compounds for internal and surrogate standards. TestAmerica Pittsburgh uses the internal standards and surrogates found in Tables 6 and 7.

18.7 When informed that the samples are from a potential chlorinated site, residual chlorine will be checked using total residual chlorine strips. If residual chlorine is detected, the Project Manager will be immediately informed and corrective action will be initiated.

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Analysis of Volatile Organics by Method 624
Based on Methods 8260B and 624

Table A-1.
Method 624 Analytes and Reporting Limits

Analytes	CAS Number	µg/L
Benzene	71-43-2	1
Bromodichloromethane	75-27-4	1
Bromoform	75-25-2	1
Bromomethane	74-83-9	1
Carbon tetrachloride	56-23-5	1
Chlorobenzene	108-90-7	1
Chloroethane	75-00-3	1
2-Chloroethyl vinyl ether *	110-75-8	2
Chloroform	67-66-3	1
Chloromethane	74-87-3	1
Dibromochloromethane	124-48-1	1
1,2-Dichlorobenzene	95-50-1	1
1,3-Dichlorobenzene	541-73-1	1
1,4-Dichlorobenzene	106-46-7	1
1,1-Dichloroethane	75-34-3	1
1,2-Dichloroethane	107-06-2	1
1,1-Dichloroethene	75-35-4	1
trans-1,2-Dichloroethene	156-60-5	1
1,2-Dichloropropane	78-87-5	1
cis-1,3-Dichloropropene	10061-01-5	1
trans-1,3-Dichloropropene	10061-02-6	1
Ethylbenzene	100-41-4	1
Methylene chloride	75-09-2	1
1,1,2,2-Tetrachloroethane	79-34-5	1
Tetrachloroethene	127-18-4	1
Toluene	108-88-3	1
1,1,1-Trichloroethane (1,1,1-Trichloroethene)	71-55-6	1
1,1,2-Trichloroethane (1,1,2-Trichloroethene)	79-00-5	1
Trichloroethene (Trichloroethane)	79-01-6	1
Trichlorofluoromethane	75-69-4	1
Vinyl chloride	75-01-4	1

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* 2-Chloroethylvinyl ether degrades under acidic conditions and cannot be determined in an acid preserved sample.

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Analysis of Volatile Organics by Method 624
Based on Methods 8260B and 624

Table A-2.
Method 624 QC Acceptance Criteria

Analytes	Daily QC Check acceptance criteria %Recovery	Mean recovery, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Standard deviation, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Matrix spike acceptance criteria (% Recovery)
Benzene	64-136	15.2-26.0	6.9	37-151
Bromodichloromethane	65-135	10.1-28.0	6.4	35-155
Bromoform	71-129	11.4-31.1	5.4	45-169
Bromomethane	14-186	D-41.2	17.9	D-242
Carbon tetrachloride	73-127	17.2-23.5	5.2	70-140
Chlorobenzene	66-134	16.4-27.4	6.3	37-160
Chloroethane	38-162	8.4-40.4	11.4	14-230
2-Chloroethyl vinyl ether	0-224	D-50.4	25.9	D-305
Chloroform	67-133	13.7-24.2	6.1	51-138
Chloromethane	D-204	D-45.9	19.8	D-273
Dibromochloromethane	67-133	13.8-26.6	6.1	53-149
1,2-Dichlorobenzene	63-137	11.8-34.7	7.1	18-190
1,3-Dichlorobenzene	73-127	17.0-28.8	5.5	59-156
1,4-Dichlorobenzene	63-137	11.8-34.7	7.1	18-190
1,1-Dichloroethane	72-128	14.2-28.5	5.1	59-155
1,2-Dichloroethane	68-132	14.3-27.4	6.0	49-155
1,1-Dichloroethene	50-150	3.7-42.3	9.1	D-234
trans-1,2-Dichloroethene	69-131	13.6-28.5	5.7	54-156
1,2-Dichloropropane	34-166	3.8-36.2	13.8	D-210
cis-1,3-Dichloropropene	24-176	1.0-39.0	15.8	D-227
trans-1,3-Dichloropropene	50-150	7.6-32.4	10.4	17-183
Ethylbenzene	59-141	17.4-26.7	7.5	37-162
Methylene chloride	60-140	D-41.0	7.4	D-221
1,1,2,2-Tetrachloroethane	60-140	13.5-27.2	7.4	46-157
Tetrachloroethene	73-127	17.0-26.6	5.0	64-148
Toluene	74-126	16.6-26.7	4.8	47-150
1,1,1-Trichloroethane (1,11-Trichloroehene)	75-125	13.7-30.1	4.6	52-162
1,1,2-Trichloroethane (1,1,2-Trichloroethene)	71-129	14.3-27.1	5.5	52-150

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Analysis of Volatile Organics by Method 624

Based on Methods 8260B and 624

Analytes	Daily QC Check acceptance criteria %Recovery	Mean recovery, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Standard deviation, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Matrix spike acceptance criteria (% Recovery)
Trichloroethene (Trichloroethane)	66-134	18.6-27.6	6.6	71-157
Trichlorofluoromethane	48-152	8.9-31.5	10.0	17-181
Vinyl chloride	4-196	D-43.5	20.0	D-251

D = MDL for the particular analyte

Note: These limits are based on method 624. The QC check acceptance criteria in percent recovery is calculated from the concentration range given in the method where the QC sample concentration is at 20 ug/L. For instance for Benzene the method states a concentration range of 12.8-27.2 ug/L. 12.8/20 *100 = 64 and 27.2/20 * 100 = 136, therefore these conversions in percent recovery is listed in the above table.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Title: Extraction and Cleanup of Organic Compounds from Waters and Solids

Method(s): SW846 3500 Series, 3600 Series, 8151A and EPA 600 Series Methods

Approvals (Signature/Date):

Brian A. Pino	Steve Jackson
10/17/07	10/16/07
Brain Pino Date	Steve Jackson Date
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10/16/07	10/16/07
Nasreen DeRubeis Date	Larry Matko Date
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This SOP was previously identified as SOP No. PITT-OP-0001, Rev. 9.

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1 SCOPE AND APPLICATION

This SOP describes procedures for preparation (extraction and cleanup) of semivolatile organic analytes in aqueous, TCLP leachate, soil, sediment, tissue and wipe matrices for analysis by Gas Chromatography (GC), Gas Chromatography / Mass Spectrometry (GC/MS), and High Performance Liquid Chromatography (HPLC). The procedures are based on SW-846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1 Extraction procedures for the following determinative methods are covered:

8081A, 8082, 8141A, 8151A, 8270C (including SIM), 8310, 608, 610, and 625

1.1.1 For methods 608 and 610, which are only applicable to aqueous matrices, only the separatory funnel extraction procedure applies.

1.1.2 For sediment samples being analyzed in support of Dredged Material Management programs, method modifications are often necessary, to compensate for the high moisture content, to meet project goals. This may include increased sample weight or decreased final extract volumes. Typically these volume modifications are up to a factor of 2.

1.2 The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

1.3 For DoD requirements, refer to DoD SOP, PITT-QA-DoD-0001, Implementation of the DoD QSM Versions 3, January 2006.

2 SUMMARY OF METHOD

2.1 Separatory Funnel Extraction

A measured volume of sample, typically 1 liter, is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.2 Continuous Liquid/Liquid Extraction

A measured volume of sample, typically 1 liter, is placed into a continuous liquid/liquid extractor, adjusted, if necessary, to a specific pH and extracted with methylene chloride for 18-24 hours.

2.3 Sonication Extraction

Low level: A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent extracted three times using an ultrasonic horn. High level: A 2 g sample is mixed with anhydrous sodium sulfate. This is solvent extracted once with a microtip ultrasonic horn.

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2.4 Soxhlet Extraction

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is extracted with refluxing solvent.

2.5 Accelerated Soxhlet (Soxtherm®) Extraction

A measured weight of sample, typically 15 g, or one whole wipe sample is mixed with anhydrous sodium sulfate and magnesium sulfate to form a free flowing powder. This is extracted with an accelerated soxhlet unit.

2.6 Cleanup and Concentration

Procedures are presented for removing interferents from sample extracts, and for drying and concentration of the extract to final volume for analysis.

2.7 Phenoxy Acid Herbicide extractions

Procedures for the extraction and cleanup of phenoxy acid herbicides are presented in Appendix A.

3 DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the Quality Management Plan (QMP).

4 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 Samples containing or suspected to contain cyanide or sulfide concentrations at or greater than 250 ppm or 500 ppm, respectively, shall be processed in a fume hood.

5.3 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

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considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed.

5.4 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.

5.5 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 KD apparatus and glass separatory funnels have ground glass joints, which can become stuck. Technicians must use Kevlar or other cut/puncture resistant gloves when separating stuck joints.

5.6 Ultrasonic disrupters can produce high intensity noise and must be used in an area with adequate noise protection.

5.7 Care must be used when separating soxhlet bodies. Protective gloves must be used when separating stuck glass joints.

5.8 Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

5.9 Mercury is a highly toxic compound that must be handled with care. Spilled mercury requires that special clean-up tools and procedures be used. Mercury is a corrosive material that will readily react with aluminum foil. Do not use aluminum foil or any aluminum products when working with mercury.

5.10 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.

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Material	Hazards	Exposure Limit[i]	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.
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 with six months.**
Florisil	**Irritant**	**TLV 10mg/m3 PEL 5mg/m3**	**May cause irritation if inhaled or adsorbed through the skin.**
Mercury	Poison	0.1 Mg/M3 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.
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[ii]	Corrosive Oxidizer Dehydrator	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.

[i] Exposure limit refers to the OSHA regulatory exposure limit.
[ii] Always add acid to water to prevent violent reactions.

5.11 Eye protection that satisfies protects against splash, laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.12 The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operations will permit. Use of methylene chloride for glassware cleaning should be avoided as far as possible.

5.13 All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to a laboratory supervisor or EH&S coordinator.

6 EQUIPMENT AND SUPPLIES

6.1 Glassware should be cleaned with soap and water, rinsed with water and dried in an oven at 400°C for at least 2 hours. Alternatively the glassware can be solvent rinsed with acetone or methanol followed by methylene chloride after the water rinse.

6.2 Equipment and supplies for extraction procedures

EQUIPMENT AND SUPPLIES	Sep .fun.	CLLE	Soni	Sox	Accel Sox.	Conc
Separatory Funnel: 2 L	√					
Separatory Funnel Rack	√					
Balance: >1400 g capacity, accurate ±1 g	√	√				
pH indicator paper, wide-range: covers extraction pH	√	√				
Graduated cylinder: 1 liter. (other sizes may be used)	√	√				
Erlenmeyer Flask or Fleaker: 125 & 300 mL (other sizes optional)	√		√			
Solvent Dispenser Pump or 100 mL Graduated Cylinder	√		√			
Continuous Liquid/Liquid Extractor		√				
Round or flat Bottom: 250, 500 mL or 1 L		√				
Boiling Chips: Contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).		√		√	√	√
Cooling Condensers		√		√	√	
Heating Mantle: Rheostat controlled		√		√	√	
Auto-timer for heating mantle		√		√	√	
Beakers: 250 & 400 mL, graduated			√	√	√	
Balance: >100 g capacity, accurate ±0.1 g			√	√	√	
Soxhlet Extractor				√		

EQUIPMENT AND SUPPLIES	Sep .fun.	CLLE	Soni	Sox	Accel Sox.	Conc
Soxtherm® Extractor Gerhardt Model S 306A					√	
Glass Thimbles					√	
Sonicator (at least 300 watts)			√			
Sonicator horn, 3/4 inch			√			
Kuderna-Danish (K-D) Apparatus: 500 mL						√
Concentrator Tube: 10 mL, attached to K-D with clips						√
Snyder Column: Three-ball macro						√
Water Bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.						√
Vials: Glass, 2 mL, 4 mL, and 10 mL capacity with Teflon®-lined screw-cap						√
Nitrogen Blowdown Apparatus						√
Nitrogen: reagent grade.						√
Culture tubes: 10 mL, 16 mmx100 mm						√
Syringe: 1 mL	√	√	√	√	√	
Phase Separation Paper	√	√	√	√	√	
Glass Wool	√	√	√	√	√	
Glass Funnel: 75 X 75 mm	√	√	√	√	√	√
Disposable Pipettes	√	√	√	√	√	√
Aluminum foil	√	√	√	√	√	√
Paper Towels	√	√	√	√	√	√
Horizon Dry Vaps						√
Dry disk separation membranes						√

6.3 Equipment and Supplies for Cleanup Procedures

EQUIPMENT AND SUPPLIES	GPC	Florisil	Sulfur	Acid
Gel permeation chromatography system (GPC Autoprep Model 1002A or 1002B Analytical Biochemical Laboratories, Inc. or Zymark Benchmate or equivalent).	√			
Bio Beads: (S-X3) -200-400 mesh, 70 gm (Bio-Rad Laboratories, Richmond, CA, Catalog 152-2750 or equivalent).	√			
Chromatographic column: 700 mm x 25 mm ID glass column. Flow is upward.	√			
Ultraviolet detector: Fixed wavelength (254 nm) and a semi-prep flow-through cell.	√			
Strip chart recorder, recording integrator, or laboratory data system.	√			
Syringe: 10 mL with Luerlok fitting.	√			
Syringe filter assembly, with disposable 5 um filter discs, Millipore No. LSWP 01300 or equivalent.	√			
Chromatographic column: 250 mm long x 10 mm ID; with Pyrex glass wool at the bottom and a Teflon stopcock (for silica gel cleanup).	√			

EQUIPMENT AND SUPPLIES	GPC	Florisil	Sulfur	Acid
Vacuum system for eluting multiple cleanup cartridges. Vac Elute Manifold - Analytichem International, J.T. Baker, or Supelco (or equivalent). The manifold design must ensure that there is no contact between plastics containing phthalates and sample extracts.		√		
Vacuum trap made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.		√		
Vacuum pressure gauge.		√		
Rack for holding 10 mL volumetric flasks in the manifold.		√		
Mechanical shaker or mixer: Vortex Genie or equivalent.			√	√
Separatory Funnels with Ground-Glass Stoppers: 250 mL				
Erlenmeyer Flasks: 125 mL				
Disposable Pipettes		√	√	√
Culture tubes: 10 mL, 16 mmx100 mm	√	√	√	√

7 REAGENTS AND STANDARDS

7.1 Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Sep fun.	CLLE	Soni	Sox	Accel. Sox.	Conc
Sodium hydroxide (NaOH), Pellets: Reagent Grade	√	√				
Sodium hydroxide solution, 10 N: Dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	√	√				
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade	√	√				
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	√	√				
Organic free reagent water.	√	√				
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	√	√			√	
Magnesium Sulfate, Anhydrous powder				√	√	
Extraction/Exchange Solvents: Methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	√	√	√	√	√	√
Acetone: Used for cleaning	√	√	√	√	√	√
50:50 Sodium Sulfate/Magnesium Sulfate			√	√	√	

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7.2 Reagents for Cleanup Procedures

REAGENTS	GPC	Florisil	Sulfur	Acid
Florisil: 500 mg or 1 g cartridges with stainless steel or Teflon frits (catalog 694-313, Analytichem, 24201 Frampton Ave., Harbor City, CA, or equivalent.)		√		
Mercury: triple distilled			√	
Tetrabutylammonium hydrogen sulfate			√	
Sodium sulfite			√	
Tetrabutylammonium (TBA) sulfite reagent: Prepare reagent by dissolving 3.39 g of Tetrabutylammonium hydrogen sulfate in 100 mL organic-free reagent water. Extract this solution 3 times with 20 mL portions of hexane. Discard the hexane extracts. Add 25 g sodium sulfite to the water solution.			√	
2-Propanol			√	
Nitric acid: 1N			√	
Copper powder: remove oxides (if powder is dark) by treating with 1N nitric acid, rinse with organic-free reagent water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.			√	
Sulfuric acid, Concentrated				√
Sodium hydroxide, Pellets				
Sodium hydroxide, 10N: Dissolve 40 g of NaOH in 100 mL of reagent water				
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade				
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.				

7.3 Standards

7.3.1 Stock Standards

Stock standards are purchased as certified solutions or prepared from neat. Semivolatile stock standards are stored at $\leq 6^{\circ}C$. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of preparation, if prepared in house, or from the time the ampule is opened if purchased.) Standards must be allowed to come to room temperature before use.

7.3.2 Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

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7.3.3 Matrix Spiking and Laboratory Control Spiking Standards.

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards. Spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.4 GPC calibration solution - prepare or purchase a solution in methylene chloride that contains the following analytes in the concentrations listed below:

Analyte	mg/mL
Corn Oil	25.0
Bis (2-ethylhexyl) phthalate	1.0
Methoxychlor	0.2
Perylene	0.02
Sulfur	0.08

NOTE: Sulfur is not very soluble in methylene chloride; however, it is soluble in warm corn oil. Therefore, one approach is to weigh out the corn oil, warm it, and transfer the weighed amount of sulfur into the warm corn oil. Mix it and then transfer into a volumetric flask with methylene chloride, along with the other calibration compounds. This standard has a lifetime of 6 months.

8 SAMPLE COLLECTION PRESERVATION, SHIPMENT AND STORAGE

8.1 Samples are not chemically preserved.

8.2 Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps except for tissue samples, which are stored frozen.

8.3 Holding Times

8.3.1 Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples, and 1 year for tissue samples.

8.3.2 For TCLP leachates, extraction is initiated within 7 days from when the leaching procedure is completed.

8.3.3 Analysis of the extracts is completed within 40 days of extraction.

9 QUALITY CONTROL

9.1 Quality Control Batch

The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate

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to process a matrix spike and sample duplicate in place of the MS/MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. For methods 608 and 610, a matrix spike is specified for every 10 samples. This will be done if the project/program requires a 10% matrix spike frequency and if sufficient sample volume is provided. See policy QA-003 for further definition of the batch.

9.2 Definition of matrix

The possible matrix types are aqueous, soil, sediment, tissue, waste, wipe and TCLP leachate.

9.3 Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria. Use of a LCS pair in place of a MS/MSD must be documented. Because subsamples cannot be taken from a wipe sample for MS/MSD analyses, wipe samples should be processed with a LCS/LCSD.

9.4 Sample count

Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.5 Method Blank

A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.5.1 Aqueous Method Blanks use 1000 mL of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.2 Solid method blanks use the same weight of sodium sulfate (acidified sodium sulfate for herbicides) as the extracted weights of the associated samples, spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.3 Method blanks for wipes consist of clean, unused gauze pads (that are the same as those used for the associated wipe samples) that are spiked with the surrogates and carried through the entire analytical procedure, including any cleanup steps.

9.5.4 TCLP method blanks use 200 mL of leachate fluid for GC/MS Semivolatiles and 100 mL for organochlorine pesticides, spiked with the surrogates. The leachate may optionally be diluted to 1000 mL with reagent water. The

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method blank goes through the entire analytical procedure, including any cleanup steps.

9.6 Laboratory Control Sample (LCS)

Laboratory Control Samples are well characterized, laboratory-generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.6.1 The LCS is made up in the same way as the method blank (See sections 9.5.1 - 9.5.3) but spiked with the LCS standard and the surrogates.

9.6.2 For the 600 series methods (608, 610, and 625), the LCS is equivalent to the QC Check Sample specified in the reference methods. For methods 608 and 610, a LCS is required for every 10 samples extracted.

9.7 Surrogates

9.7.1 Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.7.2 Each applicable sample, blank, LCS and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.8 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike.

9.9 Initial Demonstration of Capability

The initial demonstration of capability and method detection limit studies described in section 13 must be acceptable before analysis of samples may begin.

9.10 Quality Assurance Summaries

Certain clients may require specific project or program QC, which may supersede these method requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

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9.11 TestAmerica QC Program

Further details of QC and corrective action guidelines are presented in the TestAmerica QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10 PROCEDURE

Procedures for separatory funnel liquid/liquid extraction (10.2), continuous liquid/liquid extraction (10.3), sonication extraction (10.4), soxhlet extraction (10.5), accelerated soxhlet extraction (10.6), waste dilution (10.7), extract concentration (10.8), and extract cleanup (10.9) are presented in this section.

10.1 Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

10.2 Separatory Funnel Liquid/Liquid Extraction of Water Samples.

Refer to Figure 1 – Separatory Funnel Extraction flowchart.

10.2.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

10.2.2 Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, Note on the benchsheet, if there is any discrepancy.

10.2.3 The normal sample volume is approximately 1 liter. Other sample volumes may be used to obtain specific reporting limits, and reduced sample volumes, diluted to 1 liter with reagent water, may be used for very dirty samples.

10.2.4 Mark the meniscus on the 1 liter sample bottle. Spike the sample in the bottle with surrogate solution. Also spike the MS and MSD aliquots with Matrix Spike solution (Refer to Tables 3 and 4 for spike volumes). Mix well.

Note: If the sample bottle is completely full, it may be difficult to add the spike solutions to the bottle. In this case, transfer the sample to the separatory funnel and then add the spike.

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10.2.5 Sample pH is adjusted, as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

10.2.6 Transfer the entire sample to the separatory funnel. Rinse the sample bottle with 60 mL of methylene chloride and transfer to the separatory funnel.

> Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the sample container has been sealed and inverted. Vent into hood away from analysts and other samples.

10.2.7 The sample volume is determined by filling the sample bottle with reagent water up to the meniscus and measuring that volume in a graduated cylinder. Record the volume to the nearest 10 mLs.

10.2.7.1 If the entire sample bottle will not be used (i.e., for smaller sample aliquots such as TCLP), mix the sample in the bottle and measure out the desired volume in a graduated cylinder. Spike the surrogate, and MS solution, where appropriate, and adjust initial sample pH in the cylinder. Transfer the aliquot to the separatory funnel.

10.2.7.2 Rinse the cylinder with 60 mL of methylene chloride and transfer to the separatory funnel.

10.2.8 Prepare a method blank and LCS for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS. The LCS is spiked with the surrogate and matrix spike solutions, the method blank only with the surrogates (see Tables 3 and 4 for spike volumes).

10.2.9 Use 100 mL of leachate for TCLP pesticides, and 200 mL of leachate for TCLP semivolatiles, measured in a graduated cylinder. The leachate may be made up to 1 L in volume with reagent water.

10.2.10 For a TCLP method blank, measure 100 mL (pesticides) or 200 mL (semivolatiles) of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Add 60 mL of methylene chloride to the separatory funnel. The TCLP leachate may be diluted to approximately 1 liter before extraction, if desired.

10.2.11 Seal and shake or rotate the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure.

> Warning: Dichloromethane 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.

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10.2.12 Allow the organic layer to separate from the water phase until complete visible separation has been achieved (approximately 10 minutes). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. If the emulsion cannot be broken (recovery of <80% of the methylene chloride*), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous liquid-liquid extractor (CLLE) and proceed as described in continuous liquid-liquid extraction (Section 10.3). If this is done, the sample must be extracted as part of a valid CLLE batch.

*Note: 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.

10.2.13 Fill a funnel with 10-20 g of anhydrous sodium sulfate. The funnel can be plugged with glass wool or filter paper may be used to hold the sodium sulfate. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

10.2.14 Repeat the extraction process two more times using fresh 60 mL portions of solvent, combining the three solvent extracts in the collection container.

10.2.15 If extraction at a secondary pH is required, adjust the pH of the sample in the separatory funnel to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the benchsheet. Serially extract with three 60 mL portions of methylene chloride, as outlined in Steps *10.2.10 to 10.2.12.* Collect these three extracts in the same container used for the initial pH fraction.

Note: Alternatively, the acid and base fractions may be kept separate. This may be required for method 625. Separate analysis of the acid and base fractions may also be required for method 625. Individual client requirements must be checked before starting the extraction.

10.2.16 Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

10.2.17 Dispose of solvent and water remaining in the separatory funnel into the appropriate waste container.

10.2.18 Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 10.8 for concentration and Section 10.9 for cleanup.

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10.3 Continuous Liquid/Liquid Extraction from Water Samples:

Refer to Figure 2 – Continuous Liquid/Liquid Extraction flowchart.

10.3.1 Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

10.3.2 Assemble the apparatus. Add 200-300 mL of methylene chloride to the extractor body. Add 3 to 5 boiling chips to the round-bottom distilling flask.

10.3.3 Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against the leachate log. Note on the benchsheet if there is any discrepancy.

10.3.4 Mark the meniscus on the 1 liter sample bottle. Spike the sample in the bottle with surrogate solution. Also spike the MS and MSD aliquots with Matrix Spike solution (see Tables 3 and 4 for spike volumes). Mix well.

Note: If the sample bottle is completely full, it may be difficult to add the spike solutions to the bottle. In this case, transfer the sample to the extractor and then add the spike.

10.3.5 Sample pH is adjusted, as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

10.3.6 Transfer the entire sample to the liquid-liquid extractor. Rinse the sample bottle with 60 mL of methylene chloride and transfer to the liquid-liquid extractor.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the sample container has been sealed and inverted. Vent into hood away from analysts and other samples.

10.3.7 The sample volume is determined by filling the sample bottle with reagent water up to the meniscus and measuring that volume in a graduated cylinder. Record the volume to the nearest 10 mLs.

10.3.7.1 If the entire sample bottle will not be used (i.e., for smaller sample aliquots such as TCLP), mix the sample in the bottle and measure out the desired volume in a graduated cylinder. Spike the surrogate, and MS solution, where appropriate, and adjust initial sample pH in the cylinder. Transfer the aliquot to the liquid-liquid extractor.

10.3.7.2 Rinse the cylinder with 60 mL of methylene chloride and transfer to the liquid-liquid extractor.

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10.3.8 Prepare a method blank and LCS for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS. The method blank is spiked with the surrogates, the LCS and matrix spikes with the surrogates and matrix spiking solutions. Note that different spiking solutions are used for methods 625, 8270 and TCLP (see Tables 3 and 4 for spike volumes).

10.3.9 Use 100 mL of leachate for TCLP pesticides, and 200 mL of leachate for TCLP semivolatiles, measured in a graduated cylinder. The leachate may be made up to 1 L in volume with reagent water.

10.3.10 For a TCLP method blank, measure 100 mL (pesticides) or 200 mL (semivolatiles) of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Dilute to about 1 liter with reagent water.

10.3.11 Add reagent water to the extractor body until approximately 125 mL of methylene chloride is pushed over into the round-bottomed flask to ensure proper operation and solvent cycling. Attach cold condenser (about 10^{o}C). Turn on heating mantle. Inspect joints for leaks once solvent has begun cycling. Extract for 18-24 hours. (24 hours required for Method 625)

10.3.12 If extraction at a secondary pH is required, (see Table 1) turn off the heating mantle and allow the extractor to cool. Detach the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the benchsheet. If desired, the acid and base fractions may be kept separate by replacing the boiling flask with a clean flask and fresh solvent. Reattach the condenser and turn on heating mantle. Extract for 18-24 hours (24 hours for Method 625).

Note: Alternatively, the acid and base fractions may be kept separate. This may be required for method 625. Separate analysis of the acid and base fractions may also be required for method 625. Individual client requirements must be checked before starting the extraction.

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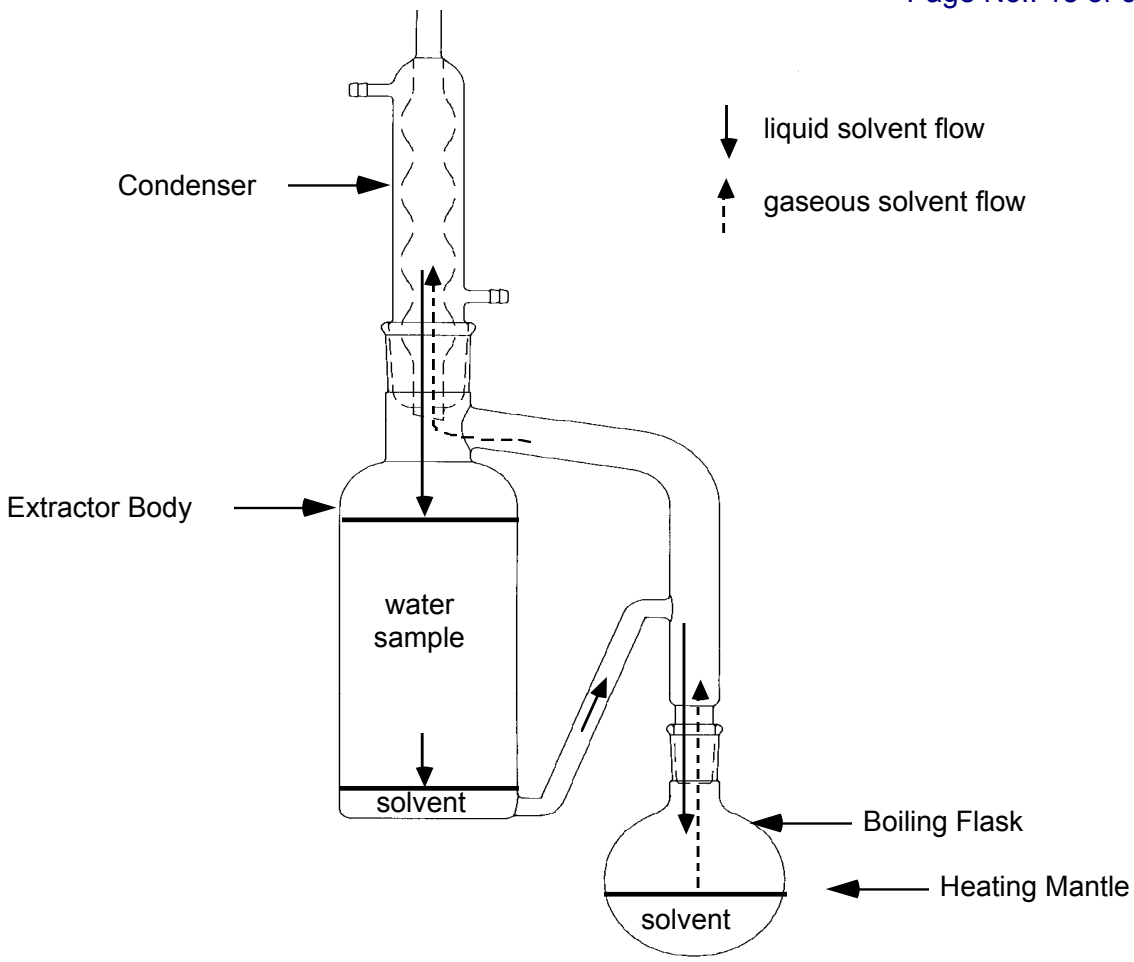

10.3.13 Turn off the heating mantle and allow the extractor to cool.

10.3.14 Place a funnel containing 10-20 g of anhydrous sodium sulfate on the Kuderna-Danish (K-D) apparatus or other glass container. The funnel can be plugged with glass wool enabling it to hold the granular anhydrous sodium sulfate or phase separation filter paper may be used.

10.3.15 Dry the extract in the round bottom flask by filtering it through the sodium sulfate filled funnel. Note that it is not necessary or advisable to attempt to add the solvent remaining in the continuous extractor body to the extract.

10.3.16 Collect the dried extract in a K-D or other glass container. Rinse the flask that contained the solvent extract with 20-30 mL of methylene chloride and add it to the funnel to complete the quantitative transfer. Dispose of solvent and water remaining in the extractor in the appropriate waste container.

Note: Some types of CLLE apparatus have built in drying columns. If this type of apparatus is used then a drying step subsequent to the extraction may not be necessary.

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10.3.17 Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 10.8 for concentration and Section 10.9 for cleanup.

10.4 Sonication

Refer to Figure 3 – Sonication Extraction flowchart.

10.4.1 Most samples will be extracted following the low-level sonication procedure. However, if high concentrations are suspected, the high-level sonication extraction procedure may be used. Both procedures are described below.

10.4.2 Decant and discard any water layer on a sediment/soil sample. Note: For sediment samples associated with most Dredged Material Management projects, the water layer is considered part of the whole sediment and should not be decanted, but re-mixed into the sample. Check project requirements before decanting any water layer. Homogenize the sample by mixing thoroughly. Tissue samples should be homogenized prior to extraction. Discard any foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager). Document if a water layer was discarded. See Tables 7 and 8 for Initial Extraction weight Adjustment for sediment samples.

10.4.3 Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

10.4.4 Low Level Procedure

10.4.5 Weigh 30 g of sample ± 1.0 g into a 250 or 400 mL beaker. Record the weight to the nearest 0.1 g in the appropriate column on the benchsheet. Use 30 g of 50:50 sodium sulfate/magnesium sulfate for the method blank and the LCS.

10.4.6 Mix weighed sample with a spatula adding enough 50:50 sodium sulfate/magnesium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

10.4.7 Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP.

10.4.8 Add surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Add the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 3 and 4 for spike volumes. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

Note: The same volume of surrogate and matrix spiking solution is used if GPC is indicated since the final volume would be reduced to compensate for loss of extract during the GPC procedure.

10.4.9 Immediately add a minimum of 100 mL of solvent to the beaker.

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Solvents:

All Tests 1:1 Methylene Chloride / Acetone

Note: Steps 10.4.5 - 10.4.9 should be performed rapidly to avoid loss of the more volatile extractables.

10.4.10 Place the bottom surface of the 3/4" horn approximately ½ inch below the surface of the solvent, but above the sediment layer.

10.4.11 Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 10 with mode switch on pulse, and percent-duty cycle knob set at 50%.

10.4.12 Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

10.4.13 Place the prepared funnel on a collection apparatus (beaker or K-D Apparatus.

10.4.14 Decant and filter extracts through the prepared funnel into a clean beaker or K-D Apparatus.

10.4.15 Repeat the extraction two more times with additional 100 mL minimum portions of solvent each time. Decant off extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone.

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

10.4.16 Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 10.8 for concentration and Section 10.9 for cleanup.

10.4.17 High Level Procedure

10.4.18 Weigh 2 g of sample into a 20 mL vial. Record the weight to the nearest 0.1 g in the appropriate column on the benchsheet. Use 2 g of sodium sulfate for the method blank and the LCS.

10.4.19 Add 2 grams of sodium sulfate to each sample and mix well.

10.4.20 Add 1 mL of surrogate to all samples including QC samples. Add 1 mL of the matrix spike solution to the LCS, MS and MSD. Depending on the test, surrogate and matrix spike solutions at higher concentrations may need to be prepared. If necessary, the preparation of these solutions will be documented in the standards database.

10.4.21 Add 8.0 mL of extraction solvent (7.0 mL to the LCS, MS, MSD) so that the final volume is 10.0 mL. The extraction solvent is as follows:

10.4.21.1 For organochlorine pesticides, organophosphorus pesticides, and PCBs (Aroclors and congeners), the solvent is hexane.

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10.4.21.2 For PAHs by HPLC, the solvent is acetonitrile.

10.4.21.3 For GC/MS semivolatiles, the solvent is methylene chloride.

10.4.22 Place the bottom surface of a 1/8" tapered microtip attached to a 1/2" horn approximately ½ inch below the surface of the solvent, but above the solid layer.

10.4.23 Sonicate each sample for 2 minutes. If the W-380 or W-385 sonicator is used, the output should be set at 10 with mode switch on pulse, and the percent-duty cycle knob set at 100% full power.

10.4.24 Loosely pack a disposable Pasteur pipette with 2 to 3 cm of glass wool. Filter the extract through the glass wool into a suitable container.

10.4.24.1 If the samples do not require cleanups or additional concentration, than the extract is ready for analysis

10.4.24.2 If cleanups (10.9) or additional concentration (10.8) are required, collected a standard volume (i.e., 5.0 mL, which represents ½ of the extract). Either account for the "loss" of half of the extract in the final sample calculations, or concentrate the extract to ½ of the standard final volume to compensate for the loss.

10.4.25 Sonicator Tuning.

10.4.25.1 Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed.

10.5 Soxhlet

Refer to Figure 4 – Soxhlet Extraction flowchart.

10.5.1 Decant and discard any water layer on a sediment/soil sample. Note: For sediment samples associated with most Dredged Material Management projects, the water layer is considered part of the whole sediment and should not be decanted, but re-mixed into the sample. Check project requirements before decanting any water layer. Homogenize the sample by mixing thoroughly. Tissue samples should be homogenized prior to extraction. Discard any foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials consult with the client. Document on benchsheet if a water layer was discarded.

10.5.2 Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

10.5.3 Weigh 30 g of sample ± 1.0 g into a beaker, recording the weight to the nearest 0.1 g on the benchsheet. Use 30 g of 50:50 sodium sulfate/magnesium sulfate for the method blank and LCS. Add 30 g of 50:50 sodium sulfate/magnesium sulfate and mix well. The mixture should have a free flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet thimble, but do not pack the thimble tightly. The

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extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the soxhlet extractor is an acceptable alternative for the thimble.

10.5.3.1 Sample weights less than 30 g but over 5 g may be used if the appropriate reporting limits can be met.

10.5.4 Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate as the matrix. The weight of 50:50 sodium sulfate/magnesium sulfate used should be approximately the weight of soil used in each sample.

10.5.5 Add the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Add the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 3 and 4 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

Note: The same volume of surrogates and matrix spiking compounds is used if GPC is indicated since the final volume would be reduced to compensate for loss of extract during the GPC procedure.

10.5.6 Place approximately 250 mL of solvent into a 500 mL flat bottom flask containing one or two clean boiling chips. Attach a cold condenser (about 10°C) to the top of the extractor. Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

Solvents:

All Tests 1:1 Methylene Chloride / Acetone

10.5.7 Allow the extract to cool after the extraction is complete, and then disassemble by gently twisting the soxhlet from the flask. Dry the extract in the flask by filtering it through a sodium sulfate filled funnel.

10.5.8 Collect the dried extract in a K-D or other glass container. Rinse the flask that contained the solvent extract with 20-30 mL of methylene chloride and add it to the funnel to complete the quantitative transfer.

10.5.9 Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 10.8 for concentration and Section 10.9 for cleanup.

10.6 Accelerated Soxhlet (Soxtherm®)

Refer to Figure 5 – Accelerated Soxhlet Extraction (Soxtherm) flowchart.

10.6.1 Decant and discard any water layer on a sediment/soil sample. Note: For sediment samples associated with most Dredged Material Management projects, the water layer is considered part of the whole sediment and should not be decanted, but re-mixed into the sample. Check project requirements

before decanting any water layer. Homogenize the sample by mixing thoroughly. Tissue samples should be homogenized prior to extraction. Discard any foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials consult with the client. Document on benchsheet if a water layer was discarded. For wipe samples, the entire contents of the original sample container will be extracted (i.e., no subsample will be taken) following the procedure for solid samples.

10.6.2 Remove surrogate and matrix spiking solutions from the refrigerator and allow to return to room temperature.

10.6.3 Weigh 15 g of sample ± 0.5 g into a beaker, recording the weight to the nearest 0.1 g on the benchsheet. Use 15 g of 50:50 sodium sulfate/magnesium sulfate for the method blank and LCS. Add 15 g of anhydrous 50:50 sodium sulfate/magnesium sulfate and mix well. The mixture should have a free flowing texture. If not, add more sodium sulfate. Add the sample/50:50 sodium sulfate/magnesium sulfate mixture to a soxhlet thimble, but do not pack the thimble tightly. The extraction thimble must drain freely for the duration of the extraction period. A glass wool plug above and below the sample in the thimble is required.

10.6.3.1 Sample weights less than 15 g but over 5 g may be used if the appropriate reporting limits can be met.

10.6.4 Prepare a method blank, LCS and MS/MSD for each batch as specified in Section 9 of this SOP, using sodium sulfate as the matrix. Use a new, clean gauze pad as the blank matrix for wipe samples and follow the procedure for extraction of solid samples. The weight of 50:50 sodium sulfate/magnesium sulfate used should be approximately the weight of soil used in each sample.

10.6.5 Add the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Add the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 3 and 4 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

> **Note:** The same volume of surrogates and matrix spiking compounds is used if GPC is indicated since the final volume would be reduced to compensate for loss of extract during the GPC procedure.

10.6.6 Place thimble in beaker containing clean boiling chips and add approximately 140 mL of solvent (see below). Place beakers into positions on the accelerated soxhlet unit. Run the appropriate program for the extraction solvent. Periodically, check the system for leaks at the joints.

10.6.6.1 For organochlorine pesticides, organophosphorus pesticides, and PCBs (Aroclors and congeners), the extraction solvent is 1:1 hexane/acetone except

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if GPC cleanup is being done. If GPC cleanup is being done, the extraction solvent is 1:1 methylene chloride/acetone.

10.6.6.2 For all other parameters, the extraction solvent is 1:1 methylene chloride/acetone.

10.6.7 Upon completion of the program, remove the beaker from the accelerated soxhlet unit and dispose of the extracted sample.

10.6.8 Collect the extract in a K-D or other glass container. Rinse the flask that contained the solvent extract with 5-10 mL of methylene chloride and add it to the funnel to complete the quantitative transfer.

10.6.9 Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 10.8 for concentration and Section 10.9 for cleanup.

10.7 Waste Dilution

10.7.1 This method is used for materials that are soluble in an organic solvent.

10.7.2 Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

10.7.3 Transfer 10 mL of the solvent to be used for dilution into a Teflon capped vial. Mark the meniscus on the vial, and then discard the solvent.

10.7.4 Tare the vial, and then transfer approximately 1g of sample to the vial. Record the weight to the nearest 0.1 g.

10.7.5 Add 1 mL of surrogate solution to each sample. Add 1 mL of matrix spike solution to the MS, MSD and LCS. Depending on the test, surrogate and matrix spike solutions at higher concentrations may need to be prepared. If necessary, the preparation of these solutions will be documented in the standards database.

10.7.6 Dilute to 10 mL with the appropriate solvent (hexane for organochlorine pesticides, organophosphorus pesticides, and PCBs (Aroclors and congeners); acetonitrile for PAHs by HPLC; methylene chloride for GC/MS semivolatiles).

10.7.7 Add 2 g + 0.1 g sodium sulfate to the sample. Cap and shake for 2 minutes.

10.7.8 Add 4-5 g sodium sulfate to a small funnel. The funnel can be plugged with glass wool or phase separation filter paper may be used to hold the sodium sulfate.

10.7.9 Pour the sample through the funnel, collecting as much as possible in a clean vial. Do NOT rinse the funnel with additional solvent, and do NOT concentrate the sample. The final volume is defined as 10 mL.

10.7.10 Label the sample, which is now ready for cleanup or analysis.

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10.8 Concentration

According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

Refer to Figure 6 – Concentration and Cleanup flowchart.

10.8.1 Kuderna-Danish (KD) Method:

10.8.1.1 Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL KD flask. For procedures where the final volume is 10 mL, a 250 mL Erlenmyer flask may be used as an alternative to the KD flask.

10.8.1.2 Add one or two clean boiling chips and the dried extract to be concentrated to the KD flask and attach a three ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column (this is important to ensure that the balls are not stuck and that the column will work properly).

10.8.1.3 Place the KD apparatus on a water bath (80-90^{o}C) so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood.

10.8.1.4 Concentrate to 5-15 mL. If the determinative method requires a solvent exchange add the appropriate exchange solvent (see Table 2) to the top of the Snyder Column, and then continue the water bath concentration back down to 1-4 mL. Refer to Table 2 for details on final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: Add an additional boiling chip with the addition of exchange solvent.

An alternative technique for solvent exchange is to replace the macro Snyder column and KD flask with a micro Snyder column, concentrate to approximately 1 mL, add 10 mL of exchange solvent, and concentrate back down to 1 mL. The extract must be cool before the macro Snyder assembly is removed.

Note: It is very important not to concentrate to dryness as analytes will be lost.

10.8.1.5 Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

10.8.1.6 If the final volume is 5 or 10 mL the extract may be made up to volume in the graduated KD tube or transferred to a 12 mL vial previously marked at the appropriate volume level. Document the final volume. Otherwise proceed to section 10.8.2

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10.8.2 Nitrogen Evaporation to Final Concentration

10.8.2.1 Transfer the entire extract to a calibrated evaporation tube. Rinse the concentrator tube with 1-2 mL of the appropriate solvent and transfer the solvent rinsate to the evaporation tube.

10.8.2.2 Place the tube in a warm water bath that is at approximately 35°C and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

10.8.2.3 During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1 mL. Concentrate the solvent accurately to the calibrated volume line and transfer the extract to the appropriate storage vial.

Note: It is very important not to concentrate to dryness as analytes will be lost.

10.8.2.4 An alternative technique is to follow the previous steps concentrating the solvent to slightly below the required final volume and then drawing the extract into a syringe. Rinse the evaporation tube with a small amount of solvent and draw additional solvent into the syringe to make up the accurate final volume.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

10.9 Cleanup Techniques

Refer to Figure 6 – Concentration and Cleanup flowchart.

The following techniques may be used to remove interfering peaks, and /or to remove materials that may cause column deterioration and/ or loss of detector sensitivity.

Gel Permeation Chromatography (Section 10.9.1) is a generally applicable technique, which can be used to prepare extracts for Semivolatiles (8270), PAHs (8310), Organochlorine pesticides (8081A), PCBs (8082), and Organophosphorus Pesticides (8141A) analysis. It is capable of separating high molecular weight material from the sample analytes, and so is particularly useful if tissue or vegetable matter is part of the sample, and for many soil samples.

Florisil column cleanup (Section 10.9.2) is particularly useful for cleanup of Organochlorine pesticides (8081A/608) and PCB (8082/608) analyses, and should normally be applied to these samples unless the matrix is clean. It separates compounds with a different polarity from the target analytes.

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Gel Permeation Chromatography and Florisil column cleanup may both be applied to samples. In this case the GPC should be performed first.

Sulfur cleanup (Section 10.9.3) is generally applied to samples for analysis by methods 8081A, 8082, and 608 since the Electron Capture Detector responds strongly to sulfur. It is performed after GPC and Florisil cleanup, if performed.

Sulfuric acid cleanup (Section 10.9.4) is applied to samples requiring analysis for PCBs (Aroclors and congeners) only. Most organic matter is destroyed by the sulfuric acid.

WARNING: Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

10.9.1 Gel Permeation Chromatography (GPC)

Note: GPC systems include the GPC Autoprep Model 1002A or 1002B Analytical Biochemical Laboratories, Inc., or equivalent.

10.9.1.1 GPC Column Preparation

10.9.1.1.1 Weigh out 70 g of Bio Beads (SX-3) into a 400-mL beaker.

10.9.1.1.2 Add approximately 300 mL of methylene chloride and stir gently.

10.9.1.1.3 Cover with aluminum foil and allow the beads to swell for a minimum of two hours. Maintain enough solvent to sufficiently cover the beads at all times.

10.9.1.1.4 Position and tighten the outlet bed support (top) plunger assembly in the tube by inserting the plunger and turning it clockwise until snug. Install the plunger near the column end but no closer than 5 cm (measured from the gel packing to the collar).

10.9.1.1.5 Turn the column upside down from its normal position with the open end up. Place the tubing from the top plunger assembly into a waste beaker below the column.

10.9.1.1.6 Swirl the bead/solvent slurry to get a homogeneous mixture and pour the mixture into the open end of the column. Transfer as much as possible, with one continuous pour, trying to minimize bubble formation. Pour enough to fill the column. Wait for the excess solvent to drain out before pouring in the rest. Add additional methylene chloride to transfer the remaining beads and to rinse the beaker and the sides of the column. If the top of the gel begins to look dry, add more methylene chloride to rewet the beads.

10.9.1.1.7 Wipe any remaining beads and solvent from the inner walls of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

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CAUTION: Do not tighten the seal if beads are between the seal and the glass surface because this can damage the seal and cause leakage.

10.9.1.1.8 Push the plunger until it meets the gel, and then compress the column bed about 4 cm.

10.9.1.1.9 Connect the column inlet to the solvent reservoir and place the column outlet tube in a waste container. Pump methylene chloride through the column at a rate of 5 mL/min. for one hour.

10.9.1.1.10 After washing the column for at least one hour, connect the column outlet tube to the inlet side of the UV detector. Connect the system outlet to the outlet side of the UV detector. Placing a restrictor (made from a piece of capillary tubing of 1/16"OD x 10/1000"ID x 2") in the outlet tube from the UV detector will prevent bubble formation, which causes a noisy UV baseline. The restrictor will not affect the flow rate. After pumping methylene chloride through the column for an additional 1-2 hours, adjust the inlet bed support plunger until approximately 6-10 psi back-pressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

10.9.1.1.11 When the GPC column is not to be used for several days, connect the column inlet and outlet lines to prevent column drying and/or channeling. If channeling occurs, the gel must be removed from the column, re-swelled, and re-poured as described above. If drying occurs, pump methylene chloride through the column until the observed column pressure is constant and the column appears wet. Always recalibrate after column drying has occurred to verify that retention volumes have not changed.

10.9.1.2 Initial Calibration of the GPC Column

10.9.1.2.1 Before use, the GPC must be calibrated based on monitoring the elution of standards with a UV detector connected to the GPC column.

10.9.1.2.2 Pump solvent through the GPC column for 2 hours. Verify that the flow rate is 4.5-5.5 mL/min. Corrective action must be taken if the flow rate is outside this range. Record the column pressure (should be 6-10 psi) and room temperature (22°C is ideal).

Note: Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared.

10.9.1.2.3 Inject the calibration solution and retain a UV trace that meets the following requirements (See resolution calculation in section 10.9.1.7):

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- Corn oil and phthalate peaks must exhibit >85% resolution.
- Phthalate and methoxychlor peaks must exhibit >85% resolution.

- Methoxychlor and perylene peaks must exhibit >85% resolution.
- Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

10.9.1.2.4 A UV trace that does not meet the criteria in paragraph 10.9.1.2.3 indicates the need for system maintenance and/or the need for a new column.

10.9.1.2.5 Determine appropriate collect and dump cycles.

10.9.1.2.6 The calibrated GPC program for organochlorine pesticides/PCB Aroclors should dump >85% of the phthalate and should collect >95% of the methoxychlor and perylene. Use a wash time of 10 minutes.

10.9.1.2.7 For GC/MS semivolatile and PAHs by HPLC extracts, initiate a column eluate collection just before the elution of bis (2-ethylhexyl) phthalate and after the elution of the corn oil. Stop eluate collection shortly after the elution of perylene. Stop collection before sulfur elutes. Use a wash time of 10 minutes after the elution of sulfur.

10.9.1.2.7.1 For PCB Congeners and Organophosphorus pesticides, this collection window should be appropriate but needs to be verified with spike solutions containing all analytes of interest.

10.9.1.2.8 Reinject the calibration solution after appropriate dump and collect cycles have been set.

10.9.1.2.9 Measure and record the volume of collected GPC eluate in a graduated cylinder.

10.9.1.2.10 The retention times for both bis(2-ethylhexyl) phthalate and perylene must not vary more than +/- 5% between calibrations.

10.9.1.3 GPC calibration check

Check the calibration of the GPC immediately after the initial calibration and at least every 7 days thereafter, while the column is in use.

10.9.1.3.1 Inject the calibration solution, and obtain a UV trace. If the retention times of bis(2-ethylhexyl)phthalate or perylene have changed by more than $\pm$ 5% use this run as the start of a new initial calibration. Otherwise, proceed with the recovery check. Excessive retention time shifts may be caused by poor laboratory temperature control or system leaks, an unstabilized column, or high laboratory temperature causing outgassing of methylene chloride. Pump methylene chloride through the system and check the retention times each day until stabilized.

10.9.1.4 GPC Recovery Check for Organoclorine Pesticides/ PCB Aroclors

10.9.1.4.1 The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Two recovery check solutions are used. The first mixture is prepared by diluting 1.0 mL of the organochlorine pesticide matrix spiking solution (Table 6) to 10

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mL in methylene chloride. The second mixture is prepared by diluting 1 mL of the PCB Aroclor matrix spiking solution (Table 6) to 10 mL with methylene chloride.

10.9.1.4.2 Load the pesticide matrix spike mixture, the PCB mixture, and a methylene chloride blank onto the GPC using the GC dump and collect values.

Note: If the analysis is for PCB Aroclors only, then the pesticide recovery check is not necessary.

10.9.1.4.3 After collecting the GPC calibration check fraction, concentrate, solvent exchanging to hexane. Adjust the final volume to 5.0 mL, and analyze by GC/EC. Refer to concentration, section 10.8.

10.9.1.4.4 The methylene chloride blank may not exceed more than one half the reporting limit of any analyte. And if the recovery of each of the single component analytes is 80-110% and if the Aroclor pattern is the same as previously run standards, then the analyst may use the column. If the above criteria are not met, there may be a need for system maintenance.

10.9.1.5 GPC Recovery Check for All other Semivolatiles

10.9.1.5.1 The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Dilute 1.0 mL of the GC/MS semivolatiles matrix spiking solution (Table 6) to 10 mL in methylene chloride for GC/MS Semivolatiles and PAHs by HPLC. For PCB Congeners and Organophosphorus pesticides, a solution containing all analytes of interest should be prepared in 10 mL of methylene chloride.

10.9.1.5.2 Load the spike mixture and a methylene chloride blank onto the GPC using the semivolatiles dump and collect values.

10.9.1.5.3 After collecting the GPC recovery check fraction, concentrate to 0.5 mL, and analyze by GC/MS for the GC/MS Semivolatiles and PAHs by HPLC. Analyze by GC/ECD for the PCB Congeners and GC/FPD for the Organophosphorus pesticides. Refer to the concentration section 10.8.

10.9.1.5.4 Recovery of the spiked analytes should be at least 60%. The blank should not contain any analytes at or above the reporting limit. If these conditions are met the column may be used for sample analysis. Otherwise correct the contamination problem, or extend the collect time to improve recovery of target analytes.

10.9.1.6 Sample Extract Cleanup

10.9.1.6.1 Reduce the sample extract volume to 1-2 mL, then adjust to 10 mL with methylene chloride prior to cleanup. This reduces the amount of acetone in the extract. Refer to section 10.8.

10.9.1.6.2 Start the pump and let the flow stabilize for 2 hours. The solvent flow rate should be 4.5-5.5 mL/min. The ideal laboratory temperature to prevent

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outgassing of the methylene chloride is 22°C. The normal backpressure is 6-10 psi.

10.9.1.6.3 In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of a 1:1 glycerol:water solution (by visual comparison) must be diluted and loaded into several loops.

10.9.1.6.4 Samples being loaded onto the GPC should be filtered with a 5 micron (or less) filter disk. Attach a filter to a 10 mL Luerlok syringe and filter the 10 mL sample extract into the sample tube.

10.9.1.6.5 Load the filtered samples into the proper sample tubes and place on the GPC.

Note: For the GPC Autoprep Model 1002A, wash the loading port with methylene chloride after loading each sample loop in order to minimize cross contamination. This step is automated on the GPC Autoprep 1002B.

10.9.1.6.6 Set the collect, dump, and wash times determined by the calibration procedure.

10.9.1.6.7 Switch to the run mode and start the automated sequence. Process each sample using the collect and dump cycle times established by the calibration procedure.

10.9.1.6.8 Collect each sample in a suitable glass container. Monitor sample volumes collected.

10.9.1.6.9 Any samples that were loaded into 2 or more positions must be recombined.

10.9.1.6.10 Concentrate semivolatile sample extracts to 0.5 mL in methylene chloride. Refer to the concentration section 10.8.

10.9.1.6.11 Solvent exchange pesticide/PCB sample extracts into hexane and concentrate to 5.0 mL. Refer to the concentration section 10.8.

10.9.1.7 Calculations

10.9.1.7.1 Resolution

To calculate the resolution between two peaks on a chromatograph, divide the depth of the valley between the peaks by the peak height of the smaller peak being resolved and multiply by 100.

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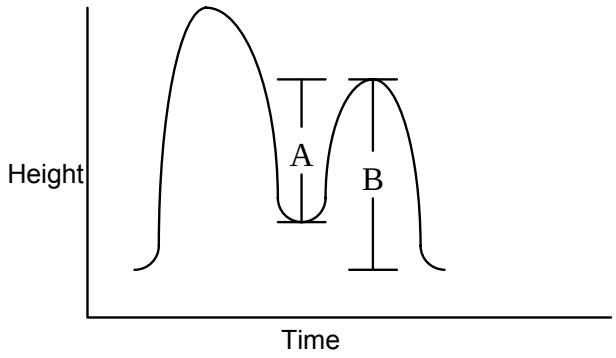
Resolution Calculation



$$\% \text{ Resolution } = \frac{A}{B} \times 100$$

Where: A = depth of valley to height of smaller peak
B = peak height of smaller peak

10.9.1.7.2 Dump Time

Mark on the chromatograph the point where collection is to begin. Measure the distance from the injection point. Divide the distance by the chart speed. Alternatively the collect and dump times may be measured by means of an integrator or data system.

$$\text{Dump time (min)} = \frac{\text{Distance (cm) from injection to collection start}}{\text{Chart speed (cm / min)}}$$

10.9.1.7.3 Collection Time

$$\text{Collection time (min)} = \frac{\text{Distance (cm) between collection start and stop}}{\text{Chart speed (cm / min)}}$$

10.9.2 Florisil Cartridge Cleanup

Florisil cleanup is generally used for organochlorine pesticides, although it may be applied to other analytes. Sections 10.9.2.1 through 10.9.2.8 outline the procedure for organochlorine pesticides, while section 10.9.2.9 outlines modifications required for other analytes.

Note 1: Systems for eluting multiple cleanup cartridges include the Supelco, Inc. Solid Phase Extraction (SPE) assembly, or equivalent.

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10.9.2.1 Before Florisil cleanup sample volume must be reduced to 10 mL (5 mL if GPC cleanup was used) and the solvent must be hexane. Refer to Section 10.8 for details of concentration.

10.9.2.2 Attach a vacuum manifold to a vacuum pump or water aspirator with a trap installed between the manifold and the vacuum. Adjust the vacuum in the manifold to 5-10 psi.

10.9.2.3 Place one Florisil cartridge into the vacuum manifold for each sample extract. Prior to cleanup of samples, pre-elute each cartridge with 5 mL of hexane/acetone (9:1). Adjust the vacuum applied to each cartridge so that the flow through each cartridge is approximately 2 mL/min. Do not allow the cartridges to go dry.

10.9.2.4 Just before the cartridges go dry, release the vacuum to the manifold and remove the manifold top.

10.9.2.5 Place a rack of clean labeled 12 mL concentrator tubes into the manifold and replace the manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.

10.9.2.6 After the clean tubes are in place, vacuum to the manifold is restored and 2.0 mL of the extract is added to the appropriate Florisil cartridge.

10.9.2.7 The organochlorine pesticides/aroclors in the extract concentrates are then eluted through the column with 8 mL of hexane/acetone (90:10) and are collected into the 10 mL culture tube or concentrator tube held in the rack inside the vacuum manifold.

10.9.2.8 Transfer the extract to a graduated concentrator tube and concentrate the extract to 2 mL. Refer to the concentration Section (10.8)

Note 1: A cartridge performance standard must be run with each lot of Florisil cartridges.

Note 2: Florisil cartridge performance check--every lot number of Florisil must be tested before use. Add 0.5 ug/mL of 2,4,5-trichlorophenol solution and 0.5 mL of Organochlorine Pesticide Calibration Standard Mix A (midpoint concentration) to 4 mL hexane. Reduce volume to 0.5 mL. Add the concentrate to a pre-washed Florisil cartridge and elute with 9 mL hexane/acetone [(90:10)(v/v)]. Rinse cartridge with 1.0 mL hexane two additional times. Concentrate eluate to 1.0 mL final volume and transfer to vial. Analyze the solution by GC/EC. The test sample must show 80 to 120% recovery of all pesticide analytes with <5% trichlorophenol recovery, and no peaks interfering with target compounds can be detected. This standard has a lifetime of six months. Alternatively, this standard may be purchased as a stock solution.

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10.9.2.9 Modifications for other analyte classes

10.9.2.9.1 PCBs

Pre-elute the cartridge with 4 mL hexane. Add 2 mL of the sample extract and elute with 3 mL hexane. The eluant will contain the PCBs together with any heptachlor, aldrin, 4,4'DDE and part of any 4,4'DDT. Any BHC isomers, heptachlor epoxide, chlordane, endosulfan I and II, endrin aldehyde and endrin sulfate and methoxychlor will be retained on the column and can be eluted in a separate fraction with 8 mL 90:10 hexane:acetone if required.

10.9.2.9.2 PAHs by GC/MS SIM (Tissue matrices only)

The extract is concentrated to approximately 2 mL. The florisil cartridge is rinsed with approximately 5 mL of methylene chloride. Discard the rinse. While collecting in a clean concentrator tube, pre-elute the cartridge with 5 mL methylene chloride and add the ~2 mL of the sample extract to the top of the cartridge. Quantitatively transfer the sample by rinsing the original vial 2-3 times with methylene chloride and add to the top of the cartridge. Once the sample extract and rinses have gravity filtered through the cartridge, add 5 mL of methylene chloride to rinse the cartridge. After the last rinse is collected, the extract is ready to be concentrated to the appropriate final volume (see Table 2).

10.9.3 Sulfur Removal

10.9.3.1 Sulfur can be removed by one of two methods: copper or tetrabutylammonium sulfite (TBA) according to laboratory preference. The TBA procedure is the laboratory default procedure. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

10.9.3.2 Sulfur Removal with Copper

10.9.3.2.1 Transfer 1.0 mL of sample extract into a centrifuge or concentrator tube.

10.9.3.2.2 Add approximately 2 g of cleaned copper powder (see 7.2 for copper cleaning procedure) to the sample extract tube.

10.9.3.2.3 Mix for one minute on a mechanical shaker.

10.9.3.2.4 If the copper changes color, sulfur was present. Repeat the sulfur removal procedure until the copper remains shiny.

10.9.3.2.5 Transfer the supernate to a clean vial.

10.9.3.3 Sulfur Removal with Tetrabutylammonium (TBA) Sulfite Reagent

10.9.3.3.1 Transfer 1.0 mL of sample extract into a culture tube.

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10.9.3.3.2 Add 1.0 mL TBA sulfite reagent and 2 mL 2-propanol to the sample extract. Cap and shake for 1 minute. If clear crystals (precipitated sodium sulfite) form, sufficient sodium sulfite is present.

10.9.3.3.3 If a precipitate does not form, add sodium sulfite in approximately 0.1 g portions until a solid residue remains after repeated shaking.

10.9.3.3.4 Add 5 mL organic free reagent water and shake for 1 minute. Allow sample to stand for 5-10 minutes. (Centrifuge if necessary to separate the layers). Transfer the sample extract (top layer) to a vial. The final volume is defined as 1.0 mL in section 10.9.3.3.1.

10.9.4 Sulfuric Acid Cleanup

10.9.4.1 Add approximately 2-5 mL of concentrated sulfuric acid to 2 mL of sample extract in a Teflon capped vial.

Caution: There must be no water present in the extract or the reaction may shatter the sample container.

10.9.4.2 Shake or vortex for about thirty seconds and allow to settle. (Centrifuge if necessary)

10.9.4.3 Remove the sample extract (top layer) from the acid using a Pasteur pipette and transfer to a clean vial. ***CAUTION:*** It is not necessary to remove all the extract since the final volume is already determined. Transfer of small amounts of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column.

10.9.4.4 If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.

10.9.4.5 Properly dispose of the acid waste.

11 CALCULATIONS / DATA REDUCTION

Not applicable.

12 METHOD PERFORMANCE

12.1 Method detection limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The procedure for the determination of the method detection limit is given in TestAmerica Pittsburgh SOP PITT-QA-0007.

12.2 Initial demonstration

Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC Check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the

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analytes of interest. The spiking level should be equivalent to a mid-level calibration. (For certain tests more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

12.2.2 Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs.

12.3 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13 POLLUTION CONTROL

13.1 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.”

13.2 Within the constraints of following the methodology in this SOP, use of organic solvents should be minimized.

14 WASTE MANAGEMENT

14.1 The following waste streams are produced when this method is carried out.

14.1.1 Methylene Chloride extraction waste. This waste is collected in waste containers identified as “Methylene Chloride Waste”, Waste #2.

14.1.2 Extracted water samples. This waste is collected in a waste container identified as “Extraction Water”, Waste #35. The bottom organic layer is drained into a container identified as “Methylene Chloride Waste”, Waste #2. The remaining aqueous layer is neutralized to a pH between 6 and 9 and discharged down lab sink/ drain.

14.1.3 Used sodium sulfate and glass wool or filter paper contaminated with methylene chloride from the extract drying step. This waste is collected in a container identified as “Lab Trash Waste”, Waste #12.

14.1.4 Assorted flammable solvent waste from various rinses. This waste is collected in waste containers identified as “Mixed Flammable Solvent Waste”, Waste 3.

14.1.5 Methylene chloride waste from various rinses. This waste is collected in waste containers identified as “Methylene Chloride Waste”, Waste #2.

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14.1.6 Miscellaneous disposable glassware contaminated with acids, caustics, solvents and sample residue. This waste is collected in a container identified as “Lab Trash Waste”, Waste #12.

15 REFERENCES

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3510C, 3520C, 3540C, 3541 3550B, 3580A 3600C, 3620B, 3640A, 3660B, and 3665A.

15.2 40 CFR Part 136, Appendix A, United States Environmental Protection Agency. 1984, Methods 608, 610, and 625.

15.2.1 PITT-QA-DoD-0001, Implementation of the DoD QSM Versions 3, January 2006.

16 ATTACHMENTS

16.1 Table 1 – Liquid/Liquid Extraction Conditions

16.2 Table 2 – Initial Volumes/Weights, Exchange Solvents and Final Volumes

16.3 Table 3 – Surrogate Spiking Solutions

16.4 Table 4 – Matrix Spike and LCS Solutions

16.5 Table 5 – Surrogate Spike Components

16.6 Table 6 – Matrix Spike Components

16.7 Table 7 – Initial Extraction Weight Adjustments for Sediment Samples (based on % Solids), Method 8270

16.8 Table 8 – Initial Extraction Weight Adjustments for Sediment Samples (based on % Solids), Methods 8081A, 8082 and 8141

16.9 Figure 1 – Separatory Funnel Extraction

16.10 Figure 2 – Continuous Liquid/Liquid Extraction

16.11 Figure 3 – Sonication Extraction

16.12 Figure 4 – Soxhlet Extraction

16.13 Figure 5 – Accelerated Soxhlet Extraction (Soxtherm)

16.14 Figure 6 – Concentration and Cleanup

16.15 Appendix A – Herbicides by Method 8151A

17 REVISION HISTORY

17.1 Revision 9, 02/01/07

17.1.1 Added wipe matrix.

17.1.2 Added procedures for low-level determinations.

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17.1.3 Changed the laboratory name to TestAmerica.

17.1.4 A number of clarifications have been made.

17.2 Revision 10, 09/24/07

17.2.1 Changed the SOP format to the new corporate SOP format.

17.2.2 Added the requirement (in Appendix A) to document the derivitization of the stock standard for herbicides by Method 8151 in the extraction log and subsequently forward it to the GC Department.

17.2.3 Added the requirement (in Sections 10.2, 10.3 and 10.7) to allow the surrogate and spiking solutions to warm to room temperature prior to adding them to the samples.

17.2.4 Made revisions to Table 8 – Initial Extraction Weight Adjustments for Sediment Samples (based on % Solids), Methods 8081A, 8082 and 8141.

17.2.5 Removed the final filtering/drying step in Section 10.6.7.

18 METHOD MODIFICATIONS

18.1 Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

18.2 Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

Table 1
Liquid /Liquid Extraction Conditions

Determinative Method	Initial Ext. pH[1]	Secondary Ext. pH[1]
BNA (8270[2]) including SIM	1-2	11-12
BNA (625)	11-12	1-2
Pesticides (8081A & 608)	5-9	None
PCB Aroclors (8082 & 608)	5-9	None
PCB Congeners (8082)	5-9	None
OP Pesticides (8141A)	as received	None
Phenols (8041)	1-2	None
PAHs (8310 & 610)	as received	None

[1] If the laboratory has validated acid only 8270C extraction (including SIM) for the target compound list required, then the base extraction step may be omitted. The required validation consists of a 4 replicate initial demonstration of capability and a method detection limit study. (See Section 13).

[2] If the laboratory has validated acid only 8270C extraction (including SIM) for the target compound list required then the base extraction step may be omitted. The required validation consists of a 4 replicate initial demonstration of capability and a method detection limit study. (See section 13).

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Table 2

Initial Volumes/Weights, Exchange Solvents, and Final Volumes[3]

Type	Initial Water Vol. (mL)	Initial Solid Wt (g)[4]	Initial Tissue Wt (g)	Exchange Solvent for Analysis	Final Volume for Analysis (mL)
Semivolatiles (8270C including SIM & 625)	1000	30 (15) NA for 625	25 NA for 625	N/A	1.0, 0.5 (low-level analyses)
Pesticides (8081A)	1000	15	6	Hexane	40.0 (waters), 20 (solids), 8.0 (tissue), 1.0 (low-level analyses)
PCB Aroclors (8082)	1000	15	6	Hexane	40.0 (waters), 20.0 (solids), 8.0 (tissue), 1.0 (low-level analyses)
Pesticide and PCB Aroclors (608)	1000	NA	NA	Hexane	8.0ml
PCB Congeners (8082)	1000	12	5	Hexane	2.0 (water) or 4.0 ml (solids and tissue)
PAH by HPLC (8310 & 610)	1000	15 NA for 610	30 NA for 610	Acetonitrile	5.0 (water), 1.0 (tissue/low-level water), 0.5 (solids)
Phenols (8041)	1000	N/A	N/A	N/A	1.0
OP Pesticides (8141A)	1000	15	12	Hexane	5.0 (water and solids) or 2.0 (tissue)

[3] Final Volumes will be ½ of the volume specified under Final Volume for Analysis if GPC Cleanup is performed (¼ if both Soxtherm® and GPC performed). GPC is required for all tissue analyses except PCBs, where it is recommended but optional if acid cleanup is performed.

[4] The values in ( ) under Initial Solid Wt. Are for the accelerated soxhlet procedures (Soxtherm®). All final volumes will be ½ of the volume listed under Final Volume for Analysis.

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Table 3
Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)[5]
BNA (8270C or 625)	100/150 ppm (20/30 ppm for low-level analyses) BNA	0.5 (water/low-level solids), 0.25 (solids), 1.0 (low-level water)
BNA (8270C SIM)	5/7.5 ppm BNA	1.0
OP Pesticides (8141A)	50 ppm Tributyl Phosphate/Triphenyl Phosphate	0.2
PAHs (8310 or 610)	10.0/20.0 ppm Benzo(e)pyrene/p-terphenyl	1.0
Pesticides (8081A)	0.8 ppm DCB/TCX	1.0 (water), 0.2 (tissue), 0.5 solids), 0.025 (low-level waters)
Pesticides and PCB Aroclors (608)	0.2 ppm DCB/TCX	0.2
Phenols (8041)	200 ppm Dibromophenol	.25
PCB Congeners (8082)	0.08 ppm TCX/BZ165	0.2 (water) or 1.0 (solids/tissue)
PCB Aroclors (8082)	0.8 ppm DCB/TCX	1.0 (water), 0.2 (tissue), 0.5 (solids), 0.025 (low-level waters)

Table 4
Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA (8270C)	100/150 ppm (20 ppm for low-level analyses) BNA	0.5 (water/low-level solids), 0.25 (solids), 1.0 (low-level water)
BNA TCLP (8270C)	BNA TCLP Spike	0.5
BNA 625	BNA NPDES Spike 100 PPM	0.5
BNA (8270C SIM)	BNA NPDES Spike-5 ppm	1.0
OP Pesticides (8141A)	10 ppm 8270 Appendix IX	0.5
PAHs (8310 or 610)	2.5/12.5 ppm PAH spike	2.0 (water), 1.0 (tissue), 0.5 (solids)
Pesticides (8081A)	1 ppm Pest	1.0, 0.5 (solids), 0.025 (low-level waters)
Pesticides TCLP (8081A)	Pest TCLP Spike	1.0
Pesticide 608	Pest NPDES Spike	0.2
PCB Congeners (8082)	0.05 ppm Congener Spike 26 compounds	0.2 (water) or 1.0 (solids/tissue)
Phenols (8041)	100 ppm Phenol Spike	0.25
PCB Aroclors 608	10 ppm PCB Spike	0.2
PCB Aroclors (8082)	40 ppm PCB Spike	1.0, 0.5 (solids), 0.025 (low-level waters)

[5] Solid samples being extracted using the Soxtherm® procedure will be spiked will ½ of the volume noted.

Table 5
Surrogate Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
BNA (8270C)	2-Fluorobiphenyl Nitrobenzene-d5 p-Terphenyl-d14 2-Fluorophenol Phenol-d6 2,4,6-Tribromophenol 1,2-Dichlorobenzene-d4 2-Chlorophenol-d4	Methanol	100, 20(low-level analyses) 100, 20(low-level analyses) 100, 20(low-level analyses) 150, 30(low-level analyses) 150, 30(low-level analyses) 150, 30(low-level analyses) 100, 20(low-level analyses) 150, 30(low-level analyses)
BNA (8270C SIM)	2-Fluorobiphenyl Nitrobenzene-d5 p-Terphenyl-d14 2-Fluorophenol Phenol-d6 2,4,6-Tribromophenol 1,2-Dichlorobenzene-d4 2-Chlorophenol-d4	Methanol	5 5 5 7.5 7.5 7.5 5 7.5
Pest/PCB Aroclors (8081A, 8082, 608)	Decachlorobiphenyl Tetrachloro-m-xylene	Acetone	0.2 0.2
Phenol (8041)	Dibromophenol	Acetone	200
PCB Congeners (8082)	BZ205 Tetrachloro-m-xylene	Acetone	0.025 0.025
PAHs (8310, 610)	Benzo(e)pyrene p-Terphenyl	Acetonitrile	10 20
OP Pesticides (8141A)	Tributyl phosphate Triphenyl phosphate	Acetone	50 50

Table 6
Matrix Spike Components

Type	Compounds	Solvent
BNA (8270C & 625)	See SOP PITT-MS-0001.	Methanol
BNA (8270C-SIM)	See SOP PITT-MS-0003	Methanol
BNA TCLP (8270C)	See SOP PITT-MS-0001	Methanol
Pesticides (8081A, 608)	See SOP PT-GC-001	Acetone
Pest TCLP (8081A)	See SOP PT-GC-001	Acetone
OP Pesticides (8141A)	See SOP PT-GC-001	Acetone
Phenol (8041)	See SOP PT-GC-001	Acetone
PAHs (8310 & 610)	See SOP PT-GC-001	Acetonitrile
PCB Congeners (8082)	See SOP PT-GC-001	Acetone
PCB Aroclors (8082 or 608)	See SOP PT-GC-001	Acetone

Table 7- Initial Extraction Weight Adjustments for Sediment Samples (based on % Solids)

Method 8270

If % Solids is:	Then the weight required to get 30 grams (dry weight) is:	Final Volume mL
≤ 54%	30.0	0.5
55-59%	27.3	0.5
60-64%	25.0	0.5
65-69%	23.1	0.5
70-74%	21.5	0.5
75-79%	20.0	0.5
80-84%	18.8	0.5
85-89%	17.7	0.5
90-94%	16.7	0.5
95-99%	15.8	0.5
100%	15.0	0.5

Table 8 - Method 8081A, 8082 and 8141

If % Solids is:	Then the weight required to get 30 grams (dry weight) is:	Final Volume 8081A/8082 (mL)	Initial and Final Volume 8141 (mL)
≤ 54%	15.0	10.0	24 g / 2.0 ml
55-59%	13.6	10.0	21.8 g / 2.0 ml
60-64%	12.5	10.0	20 g / 2.0 ml
65-69%	11.5	10.0	18.5 g / 2.0 ml
70-74%	10.7	10.0	17.1 g / 2.0 ml
75-79%	10.0	10.0	16 g / 2.0 ml
80-84%	9.38	10.0	15 g / 2.0 ml
85-89%	8.82	10.0	14.1 g / 2.0 ml
90-94%	8.33	10.0	13.3 g / 2.0 ml
95-99%	7.89	10.0	12.6 g / 2.0 ml
100%	7.5	10.0	12 g / 2.0 ml

Add 250 uL of surrogate for 8081A/8082. Add 200 mL of matrix spike for 8081. Add 250 uL of matrix spike for 8082. For 8141, add 80 uL of surrogate and 200 uL of matrix spike.

For PCB Congeners: Extract 12 grams with a 4.0 mL final volume. Add 400 uL of surrogate and matrix spike.

- 50/50 Sodium Sulfate/Magnesium Sulfate
- If multiple vessels needed, divide surrogate evenly among all vessels.

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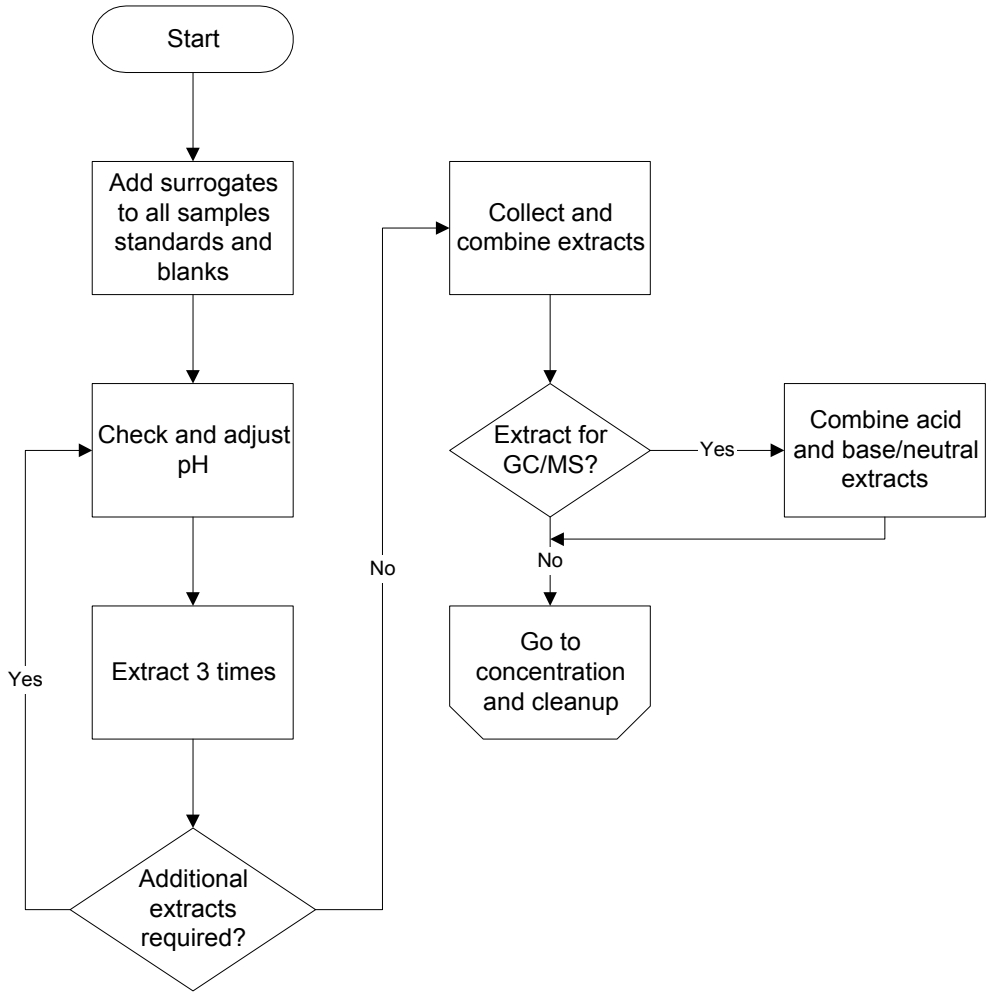
Figure 1 - Separatory Funnel Extraction



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18.2.1 Figure 2 - Continuous Liquid/Liquid Extraction

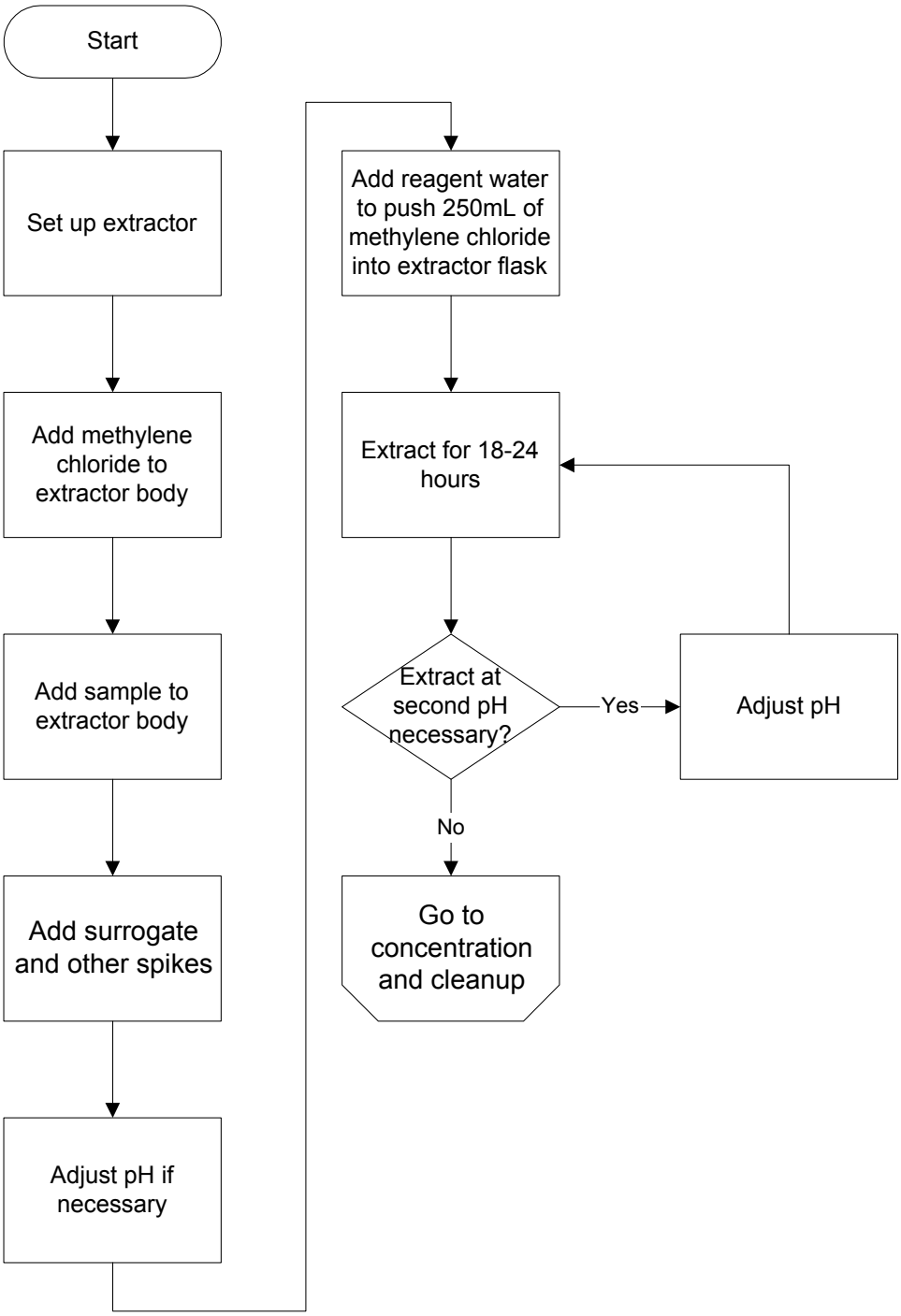
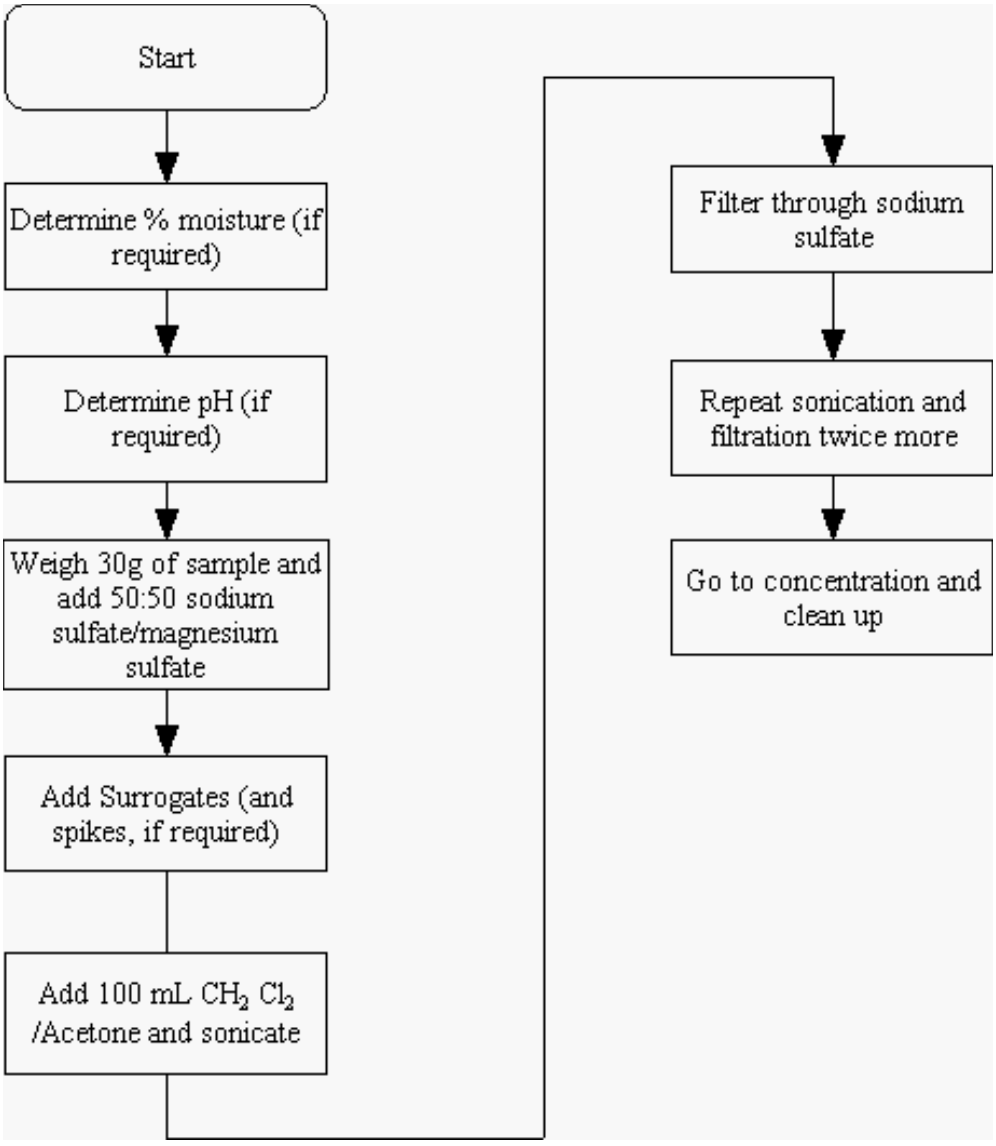
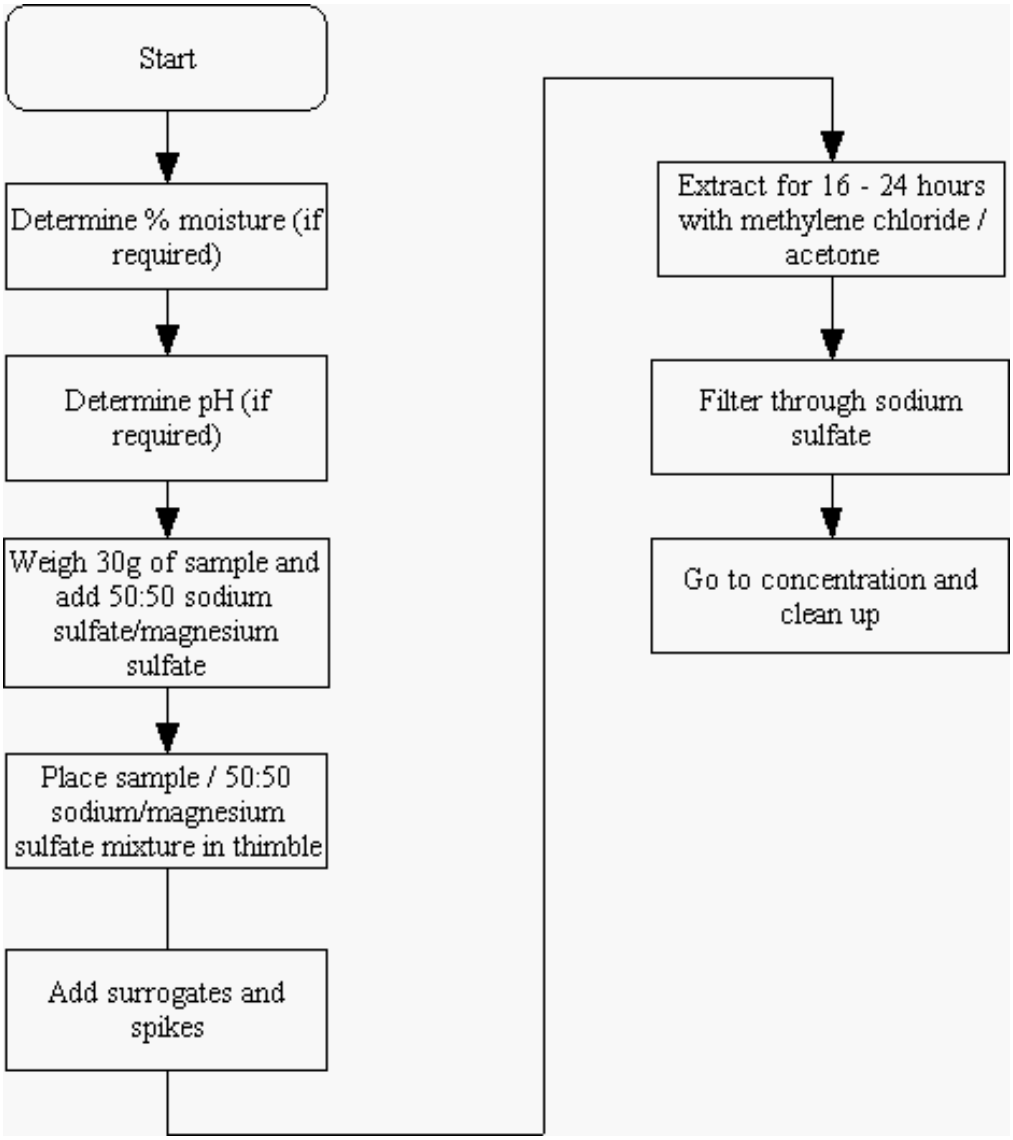


Figure 3 - Sonication Extraction



18.2.2 Figure 4 - Soxhlet Extraction



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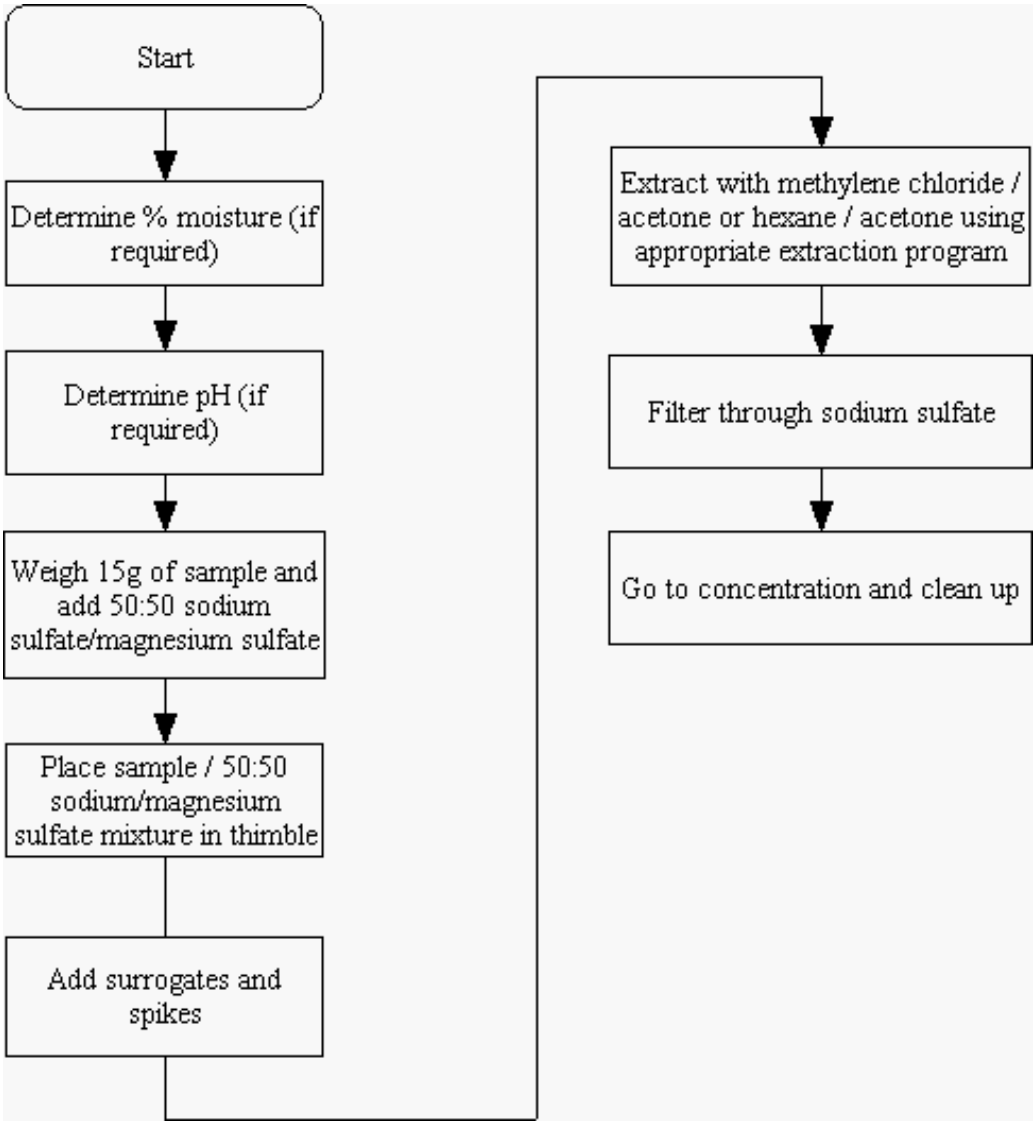
18.2.3 Figure 5 - Accelerated Soxhlet Extraction (Soxtherm®)



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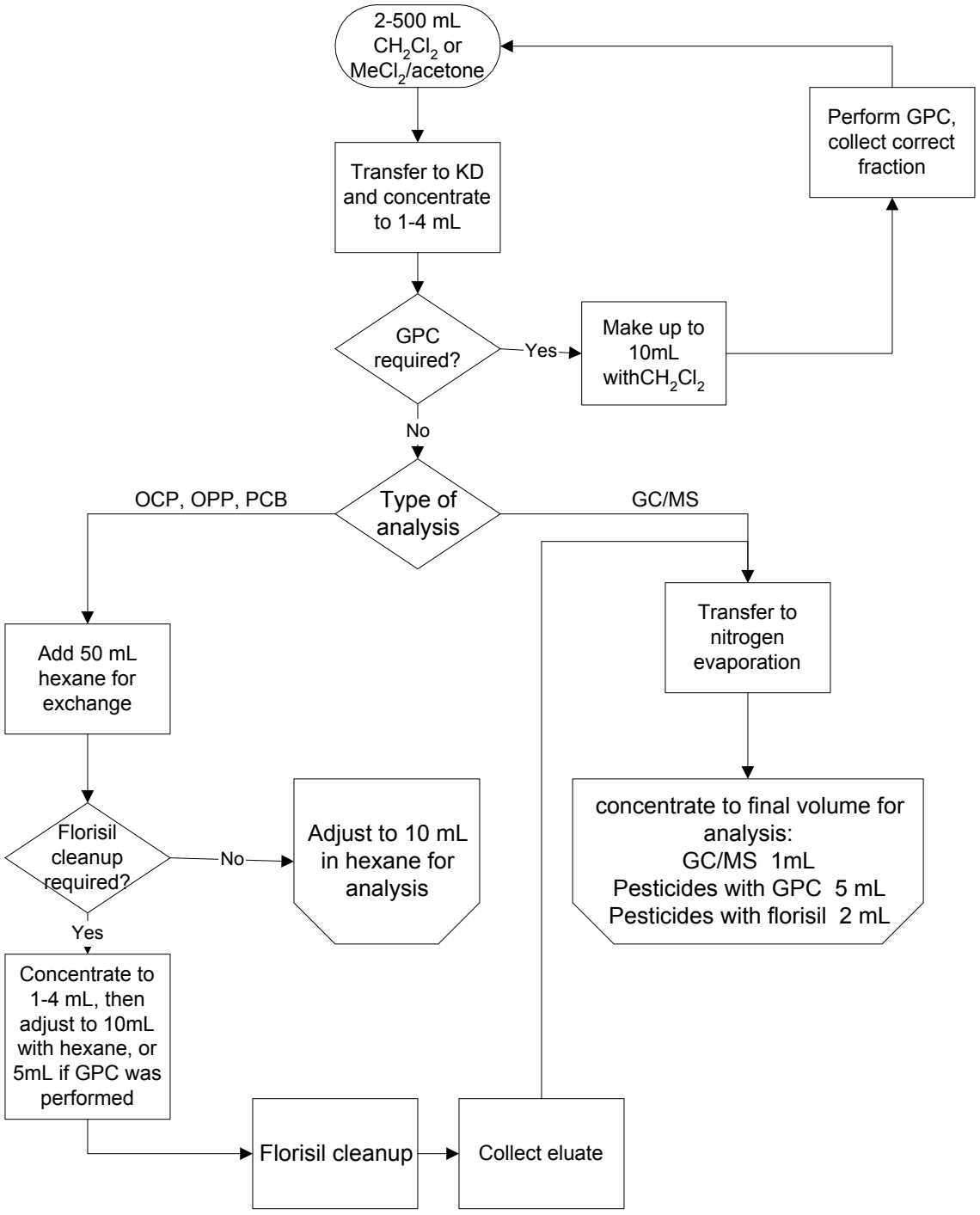
18.2.4 Figure 6 - Concentration and Cleanup



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Appendix A – Herbicides by Method 8151A

19 SCOPE AND APPLICATION

This method is applicable to the extraction of chlorinated herbicides in waters, solids, oils, and TCLP extracts. Appropriate compounds for extraction by this method are listed in PT-GC-001, Gas Chromatography of Phenoxy Acid Herbicides based on Method 8151A.

20 SUMMARY OF METHOD

This method is based on SW846 method 8151A. Aqueous samples are hydrolyzed if esters and acids are to be determined, then washed with methylene chloride by a separatory funnel extraction. After acidifying the sample the free acids are extracted into diethyl ether. Solids are extracted into methylene chloride/ acetone by sonication. If esters and acids are to be determined, the extract is hydrolyzed and extracted into diethyl ether. For both soils and aqueous samples, the free acid herbicides in the ether extract are esterified. The final volume is adjusted to prepare the extract for gas chromatography.

21 DEFINITIONS

Refer to Section 3 of the main body of this SOP.

22 INTERFERENCES

Refer to Section 4 of the main body of this SOP.

23 SAFETY

23.1 Refer to Section 5 of the main body this SOP for basic safety information.

23.2 Diethyl ether is extremely flammable. It also tends to form peroxides when exposed to air. The peroxides can present an explosion hazard, especially when the ether is concentrated.

23.3 Diethyl ether must be free of peroxides as demonstrated by EM (or equivalent) Quant test strips. This test can be done every time the ether is used or once per week if the bottle is marked with the test date(s).

23.4 Concentrated potassium hydroxide solution is highly caustic.

24 EQUIPMENT AND SUPPLIES

24.1 Refer to Section 6 of the main body of this SOP for basic extraction equipment and supplies. Additional equipment and supplies needed for this procedure are listed below.

24.2 EM Peroxide test strips

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25 REAGENTS AND STANDARDS

Reagents are listed in Section 7 of the main body of this SOP. Additional Reagents and standards needed for this procedure are listed below.

25.1 Derivitization of the stock standard must be documented in the extraction log and forwarded to the GC Department.

25.2 Reagents

25.2.1 Potassium hydroxide solution, 37% aqueous solution, (w/v): Dissolve 37 g of potassium hydroxide pellets in reagent water and dilute to 100 mL. **Caution:** Considerable heat will be generated. Other volumes of solution may be made up as convenient.

25.2.2 Sodium hydroxide solution, 6N. Dissolve 400 g NaOH in reagent water and dilute to 1.0L. **Caution:** Considerable heat will be generated. Other volumes of solution may be made up as convenient.

25.2.3 Sodium hydroxide solution, 0.1N. Dissolve 4g NaOH in reagent water and dilute to 1.0L. Other volumes of solution may be made up as convenient.

25.2.4 Sulfuric acid, 1:1 Slowly add 500 mL concentrated sulfuric acid to 500 mL water. **Caution:** Considerable heat will be generated. The acid must be added to the water. Wear protective clothing and safety glasses. Other volumes of solution may be made up as convenient.

25.2.5 Sodium sulfate, Na_2SO_4, Anhydrous, granular, acidified: Heat sodium sulfate in a shallow tray at 400°C for a minimum of 4 hours to remove phthalates and other interfering organic substances. In a large beaker, acidify by slurrying 1000 g sodium sulfate with just enough diethyl ether to cover. Add 2-5 mL of concentrated sulfuric acid and mix thoroughly. Place the mixture on a steam bath in a hood to evaporate the ether, or allow the ether to evaporate overnight. Larger or smaller batches of acidified sodium sulfate may be prepared using the reagents in the same proportions.

25.2.6 Sodium Chloride, NaCl

25.2.7 Acidified 5% sodium sulfate solution

Add 50 g of sodium sulfate to one liter of reagent water. Add 10 mL of concentrated H_2SO_4. (This reagent may be prepared in different quantities if the proportions are kept the same).

25.2.8 Diethyl ether, reagent grade.

25.2.9 Trimethylsilyldiazomethane solution (Aldrich 36,283-2)- 2.0M in hexanes (CAS # 18107-18-1).

25.2.10 Methanol, reagent grade.

25.2.11 Silicic acid

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25.3 Standards

25.3.1 Surrogate Standard

See Table A3.

25.3.2 Matrix Spike and LCS standard

See Table A4.

26 SAMPLE COLLECTION PRESERVATION, SHIPMENT AND STORAGE

26.1 Sample collection and storage is described in Section 8 of the main body of this SOP.

27 QUALITY CONTROL

27.1 Refer to Section 9 of the main body of this SOP for Quality Control procedures.

28 PROCEDURE

28.1 Preparation of Aqueous Samples

28.1.1 The glassware must be acid washed prior to use to avoid alkaline reacting with acid herbicides. Mark the meniscus on the 1 liter sample bottle. Pour the entire contents into a 2 liter separatory funnel. The sample volume is determined by filling the sample bottle with reagent water up to the meniscus and measuring the volume in a graduated cylinder (note, this is done after the bottle is rinsed with solvent). Record to the nearest 10 mL. TCLP leachates, measure 100 mL of sample in a graduated cylinder and pour into the 2 liter separatory funnel (add reagent water to bring up to approximately 1 liter).

28.1.2 Spike each sample blank, LCS and MS with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1 mL of herbicide matrix spiking solution. (Refer to Tables A1 and A2)

28.1.3 Add 250 g of NaCl to the sample and shake to dissolve the salt.

28.1.4 Hydrolysis

Use this step only if herbicide esters in addition to herbicide acids are to be determined. This is normally the case. If the herbicide esters are not to be determined, omit this step and go to 28.1.10.

Add 17 mL of 6N NaOH to the sample, seal and shake. Check the pH of the sample with pH paper. If the pH of the sample is not $\geq$12 adjust to $\geq$12 by adding more NaOH. Let the sample sit at room temperature for 2 hours to complete the hydrolysis.

28.1.5 Add 60 mL of methylene chloride to the sample bottle or graduated cylinder (TCLP samples). Rinse the bottle or graduated cylinder and add the methylene chloride to the separatory funnel.

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28.1.6 Extract the sample by shaking or rotating vigorously for 2 minutes, venting as necessary. (An automatic shaker may be used). Allow the organic layer to separate from the aqueous layer. If an emulsion layer greater than one third of the solvent layer forms, use mechanical techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool and centrifugation.

28.1.7 Discard the **methylene chloride** phase.

28.1.8 Add a second 60 mL of methylene chloride and repeat the extraction a second time, discarding the methylene chloride. Repeat the extraction a third time.

28.1.9 Add 17 mL of cold (4°C) 1:1 sulfuric acid to the sample. Seal, and shake to mix. Check the pH of the sample with pH paper. If the pH is not $\leq$ 2, add more acid to adjust the pH to $\leq$ 2.

Caution: Addition of acid may cause heat and / or pressure build up.

28.1.10 Add 120 mL diethyl ether to the sample and extract by shaking or rotating vigorously for 2 minutes, venting as necessary. (An automatic shaker may be used). Allow the organic layer to separate from the aqueous layer. If a emulsion layer greater than one third of the solvent layer forms, use mechanical techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool and centrifugation.

28.1.11 Drain the aqueous layer into a clean flask or beaker. Collect the ether phase in a clean flask or bottle containing approximately 10g of acidified anhydrous sodium sulfate.

28.1.12 Return the aqueous phase to the separatory funnel, add 60 mL diethyl ether and repeat the extraction procedure a second time, combining the ether extracts. Repeat the extraction a third time with 60 mL diethyl ether. Discard the aqueous phase after the third extraction.

28.1.13 Allow the extract to remain in contact with the sodium sulfate for at least 2 hours, shaking periodically. (May be left overnight). The drying step is critical: if the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of sodium sulfate is sufficient if some free flowing crystals are visible when the flask or bottle is swirled or shaken.

28.1.14 Proceed to Section 28.6; Concentration.

28.2 Extraction of Waste Samples

28.2.1 The glassware must be acid washed prior to use to avoid alkaline reacting with acid herbicides. Follow the Waste Dilution procedure in Section 11.7 of this SOP with the following exceptions:

- Use diethyl ether as the extraction solvent
- Use acidified sodium sulfate and acidified glasswool

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- Spike 1.0 mL of the surrogate solution to all samples and 1.0 mL of the matrix spike solution to the MS, MSD, and LCS (see Tables A1 and A2 for details).

28.2.2 Transfer 1.0 ml of the extract to a 250 mL Erlenmyer flask with a ground glass joint at the neck. Proceed to Section 28.3.14.

28.3 Extraction of soil and sediment samples

28.3.1 The glassware must be acid washed prior to use to avoid alkaline reacting with acid herbicides. Decant and discard any water layer on a sediment/soil sample. Homogenize the sample by mixing thoroughly. Discard any foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by the client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator). Document if a water layer was discarded.

28.3.2 Weigh 50.0 g of moist solid sample into a clean glass jar. Use 50 g of sodium sulfate for the Method Blank and the LCS. Acidify the sample with 5 mL of concentrated HCl.

28.3.3 There should be a small amount of liquid phase. If not, add reagent water until there is. Stir well with a spatula. (Note: This is not necessary for the method blank or LCS)

28.3.4 After 15 minutes, stir with a spatula and check the pH of the liquid phase. Add more acid if necessary to bring the pH to <2, repeating the stirring and standing time after each acid addition. (Note: The pH of the method blank and LCS are not determined.)

28.3.5 Add 60 g of acidified sodium sulfate and mix well. The sample should be free flowing. If not, add more sodium sulfate.

28.3.6 Spike each sample blank, LCS and MS with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1 mL of herbicide matrix spiking solution. (Refer to Tables A1 and A2)

28.3.7 Add a minimum of 100 mL of 1:1 methylene chloride:acetone to the beaker.

28.3.8 Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

28.3.9 Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn with mode switch on pulse, and percent-duty cycle knob set at 50%.

28.3.10 Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

28.3.11 Place the prepared funnel on a collection apparatus. If the herbicide esters are *not* to be determined, the collection apparatus is a bottle or flask

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containing approximately 10g of anhydrous acidified sodium sulfate. If the herbicide esters *are* to be determined, (normally the case) the collection apparatus is glassware suitable for the hydrolysis step, typically a KD flask.

28.3.12 Decant and filter extracts through the prepared funnel into the collection apparatus.

28.3.13 Repeat the extraction two more times with additional 100 mL minimum portions of methylene chloride / acetone each time. Decant off extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone.

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

28.3.14 If the herbicide esters are not to be determined, dry the extract as described in Section 28.5 or go to cleanup, Section 28.4. If the herbicide esters are to be determined (normally the case) proceed to Section 28.3.15.

28.3.15 Add 5 mL of 37% aqueous potassium hydroxide and 30 mL of water to the extract. Check the pH with pH paper. If the pH is not $\geq$12, adjust with additional KOH.

28.3.16 Heat on a water bath at 60-65^{o}C for 2 hours. Allow to cool. Higher temperatures, up to 90^{o}C, may be used if needed to remove the ether layer within 2 hours.

28.3.17 Transfer the solution to a separatory funnel and extract three times with 100 mL portions of methylene chloride. **Discard the methylene chloride phase.** The aqueous solution contains the herbicides.

28.3.18 Adjust the pH of the solution to $\leq$ 2 with 1:1 sulfuric acid.

28.3.19 Extract once with 40 mL diethyl ether and twice with 20 mL diethyl ether.

28.3.20 Proceed to Section 28.4, Cleanup, if required, or Section 28.5, Extract drying.

28.4 Cleanup

This cleanup step may be necessary if the procedure for determining the herbicide acids only is being followed. (See Section 28.3.14) It is not normally required if the acids and esters are being determined (the usual case). If cleanup is not required, proceed to Section 28.5, Extract drying.

28.4.1 Prepare 45 mL of basic extraction fluid by mixing 30 mL of reagent water with 15 mL of 37% KOH. Use three 15 mL portions of this fluid to partition the extract from Section 28.3.14 or 28.3.20, using a small separatory funnel. **Discard the organic phase.**

28.4.2 Adjust the pH of the solution to $\leq$ 2 with cold (4^{o}C) sulfuric acid. (1:1). Extract once with 40 mL diethyl ether and twice with 20 mL diethyl ether.

Caution: Addition of acid may cause heat and / or pressure build up.

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28.5 Extract drying

28.5.1 Combine the extracts and pour through a funnel containing acidified sodium sulfate into a flask or bottle containing approximately 10 g acidified sodium sulfate. Rinse the funnel with a little extra diethyl ether.

28.5.2 Allow the extract to remain in contact with the sodium sulfate for at least 2 hours, shaking periodically (may be left overnight). The drying step is critical: if the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of sodium sulfate is sufficient if some free flowing crystals are visible when the flask or bottle is swirled or shaken. Proceed to Section 28.6, concentration.

28.6 Concentration

28.6.1 Transfer the ether extract by decanting, or through a funnel plugged with acid washed glass wool, into a 500 mL K-D flask equipped with a 10 mL concentrator tube. Use a stirring rod to crush the caked sodium sulfate during transfer. Rinse the flask or bottle with 20-30 mL ether to complete transfer.

28.6.2 Attach a three ball Snyder column to the K-D apparatus, pre-wet the column with a few mL of ether from the top, and place the apparatus on a water bath at approximately 60°C, not to exceed 65 °C. At the proper rate of distillation, the balls of the column will chatter, but the chambers will not flood. When the apparent volume reaches 15-20 mL, remove from the water bath and allow to completely cool. Add 20 mL of hexane and concentrate to 10 mL on the water bath. Then pour the sample into a centrifuge tube, blow down to 2 ml on the nitrogen bath.

28.6.3 The extract is now ready for esterification by the trimethylsilyldiazomethane solution method (28.7).

28.7 Esterification (trimethylsilyldiazomethane solution method)

28.7.1 To the extract (hexane), add 200 uL of methanol.

28.7.2 Add 100 uL of the Trimethylsilyldiazomethane solution.

28.7.2.1 The extract should turn a yellow color. If this does not occur, add an additional 100 uL of the trimethylsilyldiazomethane solution until the yellow color persists.

28.7.3 Allow the extract to sit for 1 hour at room temperature.

28.7.4 Add approximately 0.2 g of silicic acid to each extract. Allow to stand for an additional 20 minutes.

28.7.5 Adjust the volume to 10 mL with hexane. The sample is now ready for gas chromatography.

29 CALCULATIONS / DATA REDUCTION

Not applicable.

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30 METHOD PERFORMANCE

Refer to the SOP PT-GC-001, Appendix D, for details of method performance.

31 POLLUTION CONTROL

Refer to Section 14 of the main body of this SOP.

32 WASTE MANAGEMENT

Refer to Section 15 of the main body of this SOP.

33 REFERENCES

33.1 SW-846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Chlorinated Herbicides, Method 8151A.

33.2 PITT-QA-DoD-0001, Implementation of the DoD QSM Versions 3, January 2006.

34 ATTACHMENTS

34.1 Table A1 – Herbicide Surrogate Spiking Solutions

34.2 Table A2 – Herbicide Matrix Spike and LCS Solutions

34.3 Table A3 – Herbicide Surrogate Spike Components

34.4 Table A4 – Herbicide Matrix Spike Components

34.5 Figure A1 – Extraction of Aqueous Samples

34.6 Figure A2 – Extraction of Soils and Sediments

34.7 Figure A3 – Drying, Concentration and Esterification

35 REVISION HISTORY

35.1 Revision 9, 02/01/07

35.1.1 Removed the unused diazomethane solution procedure for esterification and added the trimethylsilyldiazomethane solution esterification method.

35.1.2 Added waste extraction procedure.

35.1.3 Several clarifications have been made.

35.1.4 Removed bubbler method.

35.2 Revision 10, 09/24/07

35.2.1 Added the requirement to document the derivitization of the stock standard in the extraction log and forward to the GC Department.

36 METHOD MODIFICATIONS

36.1 Directions to add sufficient reagent water to the soil sample so that the pH can be measured have been added (Section 28.2.3)

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Table A1 Herbicide Surrogate Spiking Solutions		
Analyte Group	**Surrogate Spike Solution ID**	**Volume (mL)**
Chlorinated Acid Herbicides	Herbicides SS	1.0

Table A2 Herbicide Matrix Spike and LCS Solutions		
Analyte Group	**Matrix Spike Solution ID**	**Volume (mL)**
Chlorinated Acid Herbicides	Herbicides MS	1.0

Table A3 Herbicide Surrogate Spike Components			
Type	**Compounds[6]**	**Solvent**	**Conc. (ug/mL)**
Herbicides SS	2,4-DCAA	Methanol	10

Table A4 Herbicide Matrix Spike Components			
Type	**Compounds[7]**	**Solvent**	**Conc. (ug/mL)**
Herbicides MS	See SOP PT-GC-001	Methanol	See SOP PT-GC-001

[6] The surrogate is spiked as the free acid.

[7] The herbicide spiking solution contains the herbicides as the free acids.

Figure A1 – Extraction of Aqueous Samples

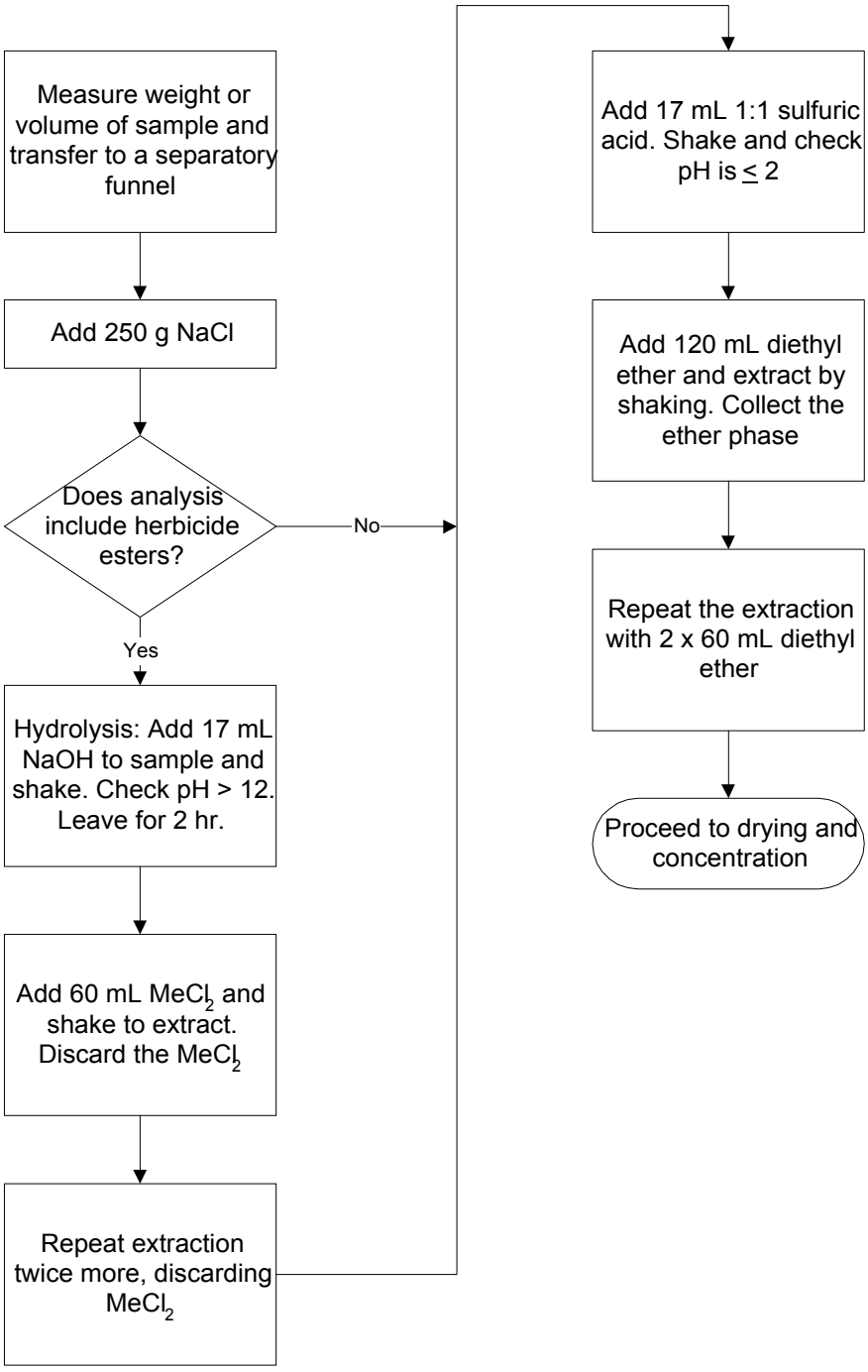

Figure A2 – Extraction of Soils and Sediments

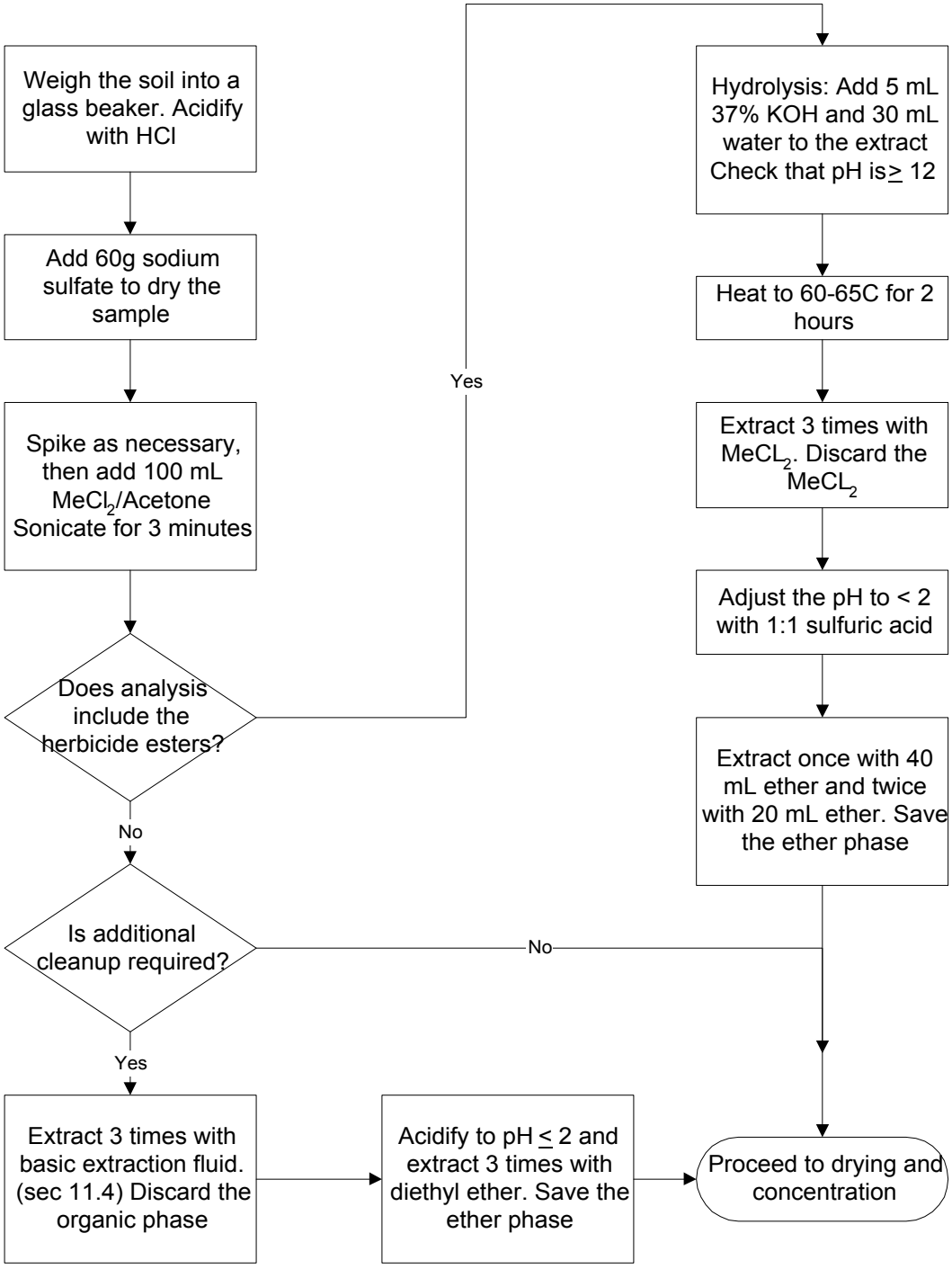

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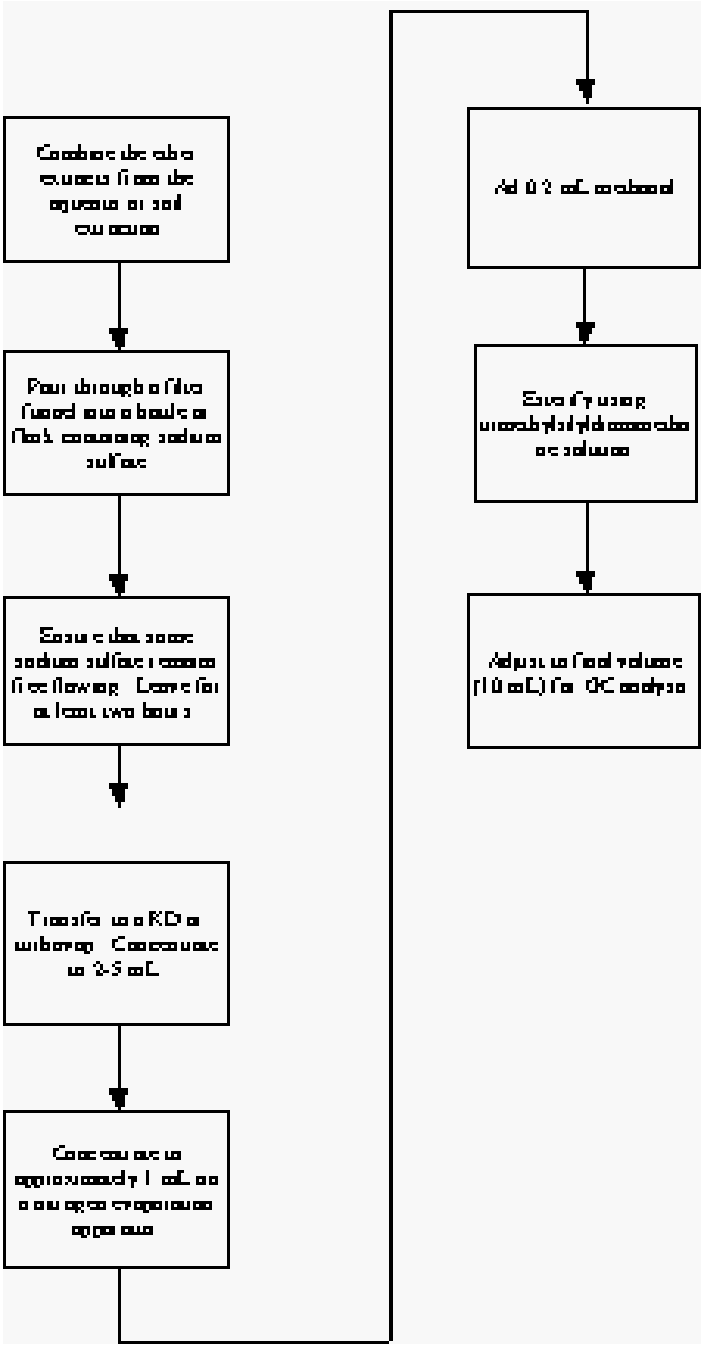
Figure A3 – Drying, Concentration and Esterification



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ATTACHMENT B-2

BLANK FIELD FORMS

CUMMINGS RITER CONSULTANTS, INC.

Date _____
Day of Week _____
No. _____
Sheet _____ of _____

FIELD ACTIVITY REPORT

Project Name:	**Project No.**
Field Activity:	
CRC Personnel On Site:	
Description on Daily Activities and Events:	

Pay Quantities:	**Project Changes/Unusual Observations:**

Weather Conditions: *a.m.* *.m.*	**Health & Safety PPE:**	**Air Monitoring Results:**
Contractor(s):		
Field Representative:		**Date:**

CUMMINGS RITER CONSULTANTS, INC.

WATER SAMPLE COLLECTION REPORT

PROJECT Koppers Pond RI SAMPLE ID _____

PROJECT NO. 502 LOCATION NO. _____

SAMPLE DATE / / SAMPLED BY _____

SAMPLE TIME (START/END) / SAMPLE SEQUENCE NO. _____

SAMPLE COLLECTION EQUIPMENT _____

FIELD MEASUREMENTS		
pH	Standard Units	
Specific Conductance	umho/cm	
Water Temperature	°	
Dissolved Oxygen	ppm	
Redox	mV	
Turbidity	NTU	

METER CALIBRATION PERFORMED? N ☐ Y ☐ DATE _____

WATER APPEARANCE, IMMISCIBLE PHASES OR ODORS: _____

SAMPLE TYPES COLLECTED

PARAMETER	VOLUME	# CONTAINERS	FIELD FILTERED?		PRESERVED?	
			Y ☐	N ☐	Y ☐ ____	N ☐
			Y ☐	N ☐	Y ☐ ____	N ☐
			Y ☐	N ☐	Y ☐ ____	N ☐
			Y ☐	N ☐	Y ☐ ____	N ☐
			Y ☐	N ☐	Y ☐ ____	N ☐
			Y ☐	N ☐	Y ☐ ____	N ☐
			Y ☐	N ☐	Y ☐ ____	N ☐
			Y ☐	N ☐	Y ☐ ____	N ☐
			Y ☐	N ☐	Y ☐ ____	N ☐
			Y ☐	N ☐	Y ☐ ____	N ☐

NUMBER OF CONTAINERS _____ FILTRATION METHOD _____

LABORATORY _____ DELIVERED VIA _____ DATE _____

WEATHER CONDITIONS _____

COMMENTS _____

CUMMINGS RITER CONSULTANTS, INC.

SEDIMENT SAMPLE FIELD COLLECTION REPORT

Project Name	Koppers Pond	Project No.	97-502
Date Collected		Time Collected	
Collected By			
	Cummings/Riter Consultants		

SAMPLE(S) LOCATION SKETCH (use reverse if necessary)

Sample I.D. No.	Depth of Sample	Sediment Description (Color, Composition, Staining, Odor, Field Measurements[1])
SD- A	0-6"	
SD- B	6-18"	
SD- C	18-30"	
SD- D	30-42"	

Sampling Method: S.S. Russian peat borer or percussion corer

Composite Sample? Y ☐ N ☒ Composite Sample I.D. No. _____

Type[2]	Volume	Per Sample?		Per Composite?	
TCL VOCs	4 oz.	Y ☒	N ☐	Y ☐	N ☐
TCL Pest/PCBs, SVOCs, pH	8 oz.	Y ☒	N ☐	Y ☐	N ☐
TOC	8 oz.	Y ☒	N ☐	Y ☐	N ☐
TAL	4 oz.	Y ☒	N ☐		
Grain Size	8 oz.	Y ☒	N ☐	Y ☐	N ☐

Number of Containers _____

Date Received by Lab _____ Laboratory: _____

Weather Conditions _____

Remarks _____

1. Organic vapor analysis, pocket penetrometer, etc.
2. Metals, VOA, organics, etc.

ANALYSIS REQUEST AND CHAIN OF CUSTODY RECORD

SEVERN TRENT **STL**

○ **STL Savannah**
5102 LaRoche Avenue
Savannah, GA 31404

Website: www.stl-inc.com
Phone: (912) 354-7858
Fax: (912) 352-0165

○ Alternate Laboratory Name/Location

Phone:
Fax:

PROJECT REFERENCE	PROJECT NO.	PROJECT LOCATION (STATE)
STL (LAB) PROJECT MANAGER	P.O. NUMBER	CONTRACT NO.
CLIENT (SITE) PM	CLIENT PHONE	CLIENT FAX
CLIENT NAME	CLIENT E-MAIL	
CLIENT ADDRESS		
COMPANY CONTRACTING THIS WORK (if applicable)		

MATRIX TYPE

REQUIRED ANALYSIS

PAGE | OF

STANDARD REPORT DELIVERY ○

DATE DUE _____

EXPEDITED REPORT DELIVERY (SURCHARGE) ○

DATE DUE _____

PRESERVATIVE

NUMBER OF COOLERS SUBMITTED PER SHIPMENT:

SAMPLE DATE	SAMPLE TIME	SAMPLE IDENTIFICATION	COMPOSITE (C) OR GRAB (G) *INDICATE*	AQUEOUS (WATER)	SOLID OR SEMISOLID	AIR	NONAQUEOUS LIQUID (OIL, SOLVENT,...)	NUMBER OF CONTAINERS SUBMITTED	REMARKS

RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RELINQUISHED BY: (SIGNATURE)	DATE	TIME
EMPTY CONTAINERS								
RECEIVED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	DATE	TIME
EMPTY CONTAINERS								

LABORATORY USE ONLY

RECEIVED FOR LABORATORY BY: (SIGNATURE)	DATE	TIME	CUSTODY INTACT YES ○ NO ○	CUSTODY SEAL NO.	STL SAVANNAH LOG NO.	LABORATORY REMARKS

APPENDIX C

HEALTH AND SAFETY PLAN

CUMMINGS RITER

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CUMMINGS RITER

APPENDIX C
HEALTH AND SAFETY PLAN
KOPPERS POND
KENTUCKY AVENUE WELLFIELD SUPERFUND SITE
OPERABLE UNIT 4
HORSEHEADS, NEW YORK

1.0 INTRODUCTION

1.1 SCOPE AND OBJECTIVES

This Health and Safety Plan (HASP) presents the procedures to be implemented for site personnel engaged in field activities during the remedial investigation (RI) for Koppers Pond at the Kentucky Avenue Wellfield Superfund Site in Horseheads, New York. The Koppers Pond Remedial Investigation/Feasibility Study (RI/FS) Group (the Group) is conducting the RI pursuant to an Administrative Settlement Agreement and Order on Consent (Index No. Comprehensive Environmental Response, Compensation, Liability Act [CERCLA]-02-2006-2025) (Settlement Agreement) entered into with the U.S. Environmental Protection Agency on September 29, 2006. In accordance with the Settlement Agreement, Koppers Pond is being addressed as Operable Unit 4 of the Kentucky Avenue Wellfield Superfund Site.

Koppers Pond consists of an approximately eight-acre, "V" shaped, warm water pond with typical water depths of approximately three to six feet. At normal stage, the surface water elevation is at 887± feet above mean sea level (msl). The pond receives inflow at the northern end of its western leg from the Industrial Drainageway, a surface water course that originates at the outlet of a 74-inch diameter underground pipe (Chemung Street Outfall) approximately 2,300 feet to the northwest. The Industrial Drainageway receives permitted process discharges originating at the former Westinghouse Electric Corporation (Westinghouse) Horseheads, New York, plant site, and the pond also receives surface runoff from a contributory watershed area of approximately 604 acres. Discharge from Koppers Pond flows in two outlet streams at its southern end, which

converge approximately 500 feet downstream to form the outlet channel. The flow in this outlet channel eventually converges with Halderman Hollow Creek, which in turn feeds into Newtown Creek, a primary tributary to the Chemung River.

The purpose of this HASP is to protect Cummings/Riter Consultants, Inc. (Cummings/ Riter), AMEC Earth & Environmental (AMEC), and other project employees and participants from potential exposures while performing activities involving potentially impacted sediment and water. The HASP also specifies measures to protect the public from site-related exposures during RI field activities.

The HASP is presented as Appendix C of the RI/FS Work Plan. The RI/FS Work Plan describes the activities to be performed, including sediment sampling, surface water sampling, potential source sampling, fish-tissue sampling, bathymetric surveying, topographic surveying, and decontamination. The elements of this HASP include procedures for personnel protection, medical surveillance program requirements, training requirements, and decontamination.

As described in the RI/FS Work Plan the field activities include the following:

- Sediment sampling for delineation of constituents of concern (COCs) in Koppers Pond, outlets, and outlet channel;
- Surface water sampling of the Industrial Drainageway, Koppers Pond, outlets, and outlet channel;
- Sampling of potential COC source areas, including permitted discharges to the Industrial Drainageway;
- Bathymetric and topographic surveying;
- Fish-tissue sampling;
- Video survey of the Chemung Street Outfall pipe;
- Sampling of floc if found in storm sewer; and
- Surveying.

The performance of these activities and other work described in the RI Work Plan will be completed as a collaborative effort among several consultants and subcontractors. Cummings/Riter will act as the general consultant for RI field activities, while AMEC will perform risk-related sampling (e.g., fish tissue) and other project activities. The HASP has been developed to address U.S. Department of Labor, Occupational Safety and Health Administration (OSHA) requirements for the entire RI project, and Cummings/Riter and AMEC have explicitly adopted this HASP to cover their respective work assignments. Other consultants and subcontractors participating in the project will be responsible for developing and implementing health and safety plans that are compliant and consistent with this HASP, or formally adopting this HASP.

1.2 TERMINOLOGY AND DEFINITIONS

The following terms are used throughout this HASP:

- **Area Monitoring:** Monitoring of airborne contamination in a work area with instruments.
- **Contamination Reduction Zone:** Area external to Exclusion Zone where contamination is removed from protective clothing.
- **Exclusion Zone:** Area where no one is allowed without protective clothing and training.
- **HSC:** Health and Safety Coordinator.
- **HSR:** Health and Safety Representative.
- **HASP:** Health and Safety Plan.
- **RI Work Plan:** Remedial Investigation Work Plan.

Any modifications to this HASP must be approved by the project Health and Safety Coordinator (HSC) as well as the project mangers for Cummings/Riter, AMEC, and any other firms adopting this HASP as their company HASP for performance of work on the Koppers Pond RI project.

2.0 PROPOSED PROJECT ORGANIZATION

The project managers and site coordinators will be responsible for seeing that site work is carried out in accordance with the procedures described in this document. The HASP will be implemented through an integrated team effort of the following key project personnel:

- Site Coordinator - Mr. Bruce Geno
- Project Health and Safety Coordinator (HSC) - Mr. Kenneth Bird
- Group Project Coordinator - Mr. Leo Brausch
- Cummings/Riter Project Manager - Mr. William Smith
- AMEC Project Manager - Mr. John Samuelian

The duties of the Project HSC are the following:

- Develop, implement, update as appropriate, and enforce this HASP;
- Coordinate with the project managers and all project participants regarding health and safety issues;
- Provide continuing health and safety support, as needed; and
- Review results of monitoring data and accident reports to formulate corrective response, as needed.

The Site Coordinator and Project HSC will designate an on-site Health and Safety Representative (HSR) based on the project team performing the RI activities. The HSR designate could change with each RI task. The HSR will have the necessary training and experience to implement this HASP. The HSR is responsible for safety at the work areas during all remedial activities. The HSR or a designated representative shall be present in the work areas during work. The HSR is responsible for assuring that all equipment calibrations and data reporting are completed in accordance with this HASP. If the provisions of this HASP are not implemented to the satisfaction of the HSR, the HSR will stop work and will not allow work to resume until corrective action has been initiated.

3.0 HAZARD EVALUATION

RI site activities will involve potential exposure to hazardous constituents and conditions. The primary RI tasks are as follows:

- Surface water sampling,
- Bathymetric surveying,
- Topographic surveying,
- Remote video surveying of the subsurface Chemung Street Outfall pipe, and
- Sediment sampling.

Cummings/Riter personnel will be involved with surface water and sediment sampling, and bathymetric surveying for the Koppers Pond RI. With respect to RI field investigations, AMEC personnel will primarily be involved in fish sampling. The personnel, equipment, and procedures specified for these activities reflect the necessary level of protection. Reclassification of activities may be performed as more environmental data are collected and evaluated. A summary of past results for Koppers Pond sediment and surface water follows.

3.1 PAST RESULTS

3.1.1 Surface Water

Historical surface water samples (1994) were collected at times when treated industrial wastewaters were being discharged from the former Westinghouse Horseheads plant, and may not be representative of current conditions. Discharge concentrations of COCs have reportedly decreased due to a reduction in operations at the former Westinghouse facility. The highest surface water concentrations detected in historical samples from the Industrial Drainageway and Koppers Pond are as follows:

- Trichloroethene (TCE) – 0.003 milligram per liter (mg/l),
- Fluoride – 7 mg/l, and
- Lead – 0.34 mg/l.

Historical measurements (2003) have indicated slightly alkaline water (geometric mean of 8.03). Metals detected in historical surface water samples reflect the influence of the permitted treated waste water discharges to the Industrial Drainageway. Trace concentrations of pesticide (α-BHC and β-BHC) were detected (<0.5 parts per billion [ppb]) in some surface water samples, and are attributed to historical area-wide or local applications.

3.1.2 Sediment

Historical sediment data associated with Koppers Pond show the presence of metals, pesticides, and polychlorinated biphenyls (PCBs) with the highest reported concentrations as follows:

- Antimony – 14 milligrams per kilogram (mg/kg)
- Arsenic – 7.8 mg/kg,
- Cadmium – 749 mg/kg,
- Chromium – 460 mg/kg,
- Copper – 960 mg/kg,
- Lead – 2,210 mg/kg,
- Nickel – 395 mg/kg,
- Mercury – 1.5 mg/kg,
- Silver – 40 mg/kg,
- PCBs – 4.5 mg/kg, and
- Pesticides – <0.01 mg/kg.

Historical sediment data have not shown significant detections of volatile organic compounds (VOCs), and relatively low concentrations of semivolatile organic compounds (SVOCs).

3.2 HAZARDS

Some safety hazards are the result of the work itself. The use of sampling and surveying equipment over open water poses potential physical hazards to workers. Maximum water depth is approximately six feet. Work is expected to be performed during non-winter months which decrease the hypothermia potential. U.S. Coast Guard-approved personal floatation devices will be worn to control this hazard. Safe operating procedures for the boat will be addressed in an initial training session.

Sampling in the storm sewer, if required, may require confined space entry. Air monitoring prior to entry will minimize potential hazards.

Samplers and surveyors are likely to encounter slippery surfaces (stream banks) and uneven ground. Proper footwear will minimize this potential hazard.

Protective equipment can impair a worker's mobility, hearing, and vision. The Level D personal protective equipment (PPE) as listed in Section 5.1 is expected to cause minimal impairment.

Site personnel will be instructed to constantly look for potential safety hazards. Project activities are not anticipated to result in off-site exposure. The disturbance of potentially impacted material will be minimal and performed in a controlled manner.

Inspection of subsurface piping will be done remotely with video equipment. No one will enter the piping or other subsurface storm water structures.

Biological hazards, including snakes, poisonous vegetation, and insects, are likely to be encountered during the RI activities. The use of PPE, including long-sleeved shirts in heavy vegetation areas, will minimize exposure. Also, decontamination after contact with insects or vegetation will reduce exposure symptoms. The use of insect repellent will aid in controlling biological hazards due to insects.

Several potentially hazardous materials, i.e., methanol, hexane, nitric acid, and Alconox® (soap), will be used during decontamination of sampling equipment. The Alconox® will be diluted with water prior to use, and small quantities of methanol, hexane, and nitric acid (less than ten milliliters) will be used for decontamination. Material Safety Data Sheets (MSDS) for these compounds are included in Attachment C-1.

The overall hazard rating for this site is low. Selection of the overall hazard rating was based on the concentration of compounds previously detected in surface water and sediment, and on the site tasks to be performed.

4.0 MEDICAL SURVEILLANCE REQUIREMENTS

Site personnel with the potential for exposure to impacted sediment or surface water are participants in a medical monitoring program in accordance with 29 Code of Federal Regulations (CFR) 1910.120. Personnel who participate in the field program will provide written copies of their certifications to the Project HSC prior to the start of the field efforts.

Medical restrictions that would inhibit personnel from performing the required work tasks are reviewed by the Project HSC when developing the task teams. Work restrictions will be incorporated into the project staffing selection process.

5.0 PERSONAL PROTECTIVE EQUIPMENT

5.1 LEVEL OF PROTECTION

Protective equipment has been selected based on the hazard evaluation and the activities performed. The level of protection by activity is as follows:

- **SURVEYING**
  Surveying from a boat will be initiated at Level D protection described below with U.S. Coast Guard-approved personal floatation devices. The boat will have sufficient capacity and stability to support the surveyors and equipment.

  Topographic (land) surveying will also be performed in Level D protection. Surveyors along shoreline or shallow water (less than two feet) shall wear boots or waders.

- **EQUIPMENT DECONTAMINATION**
  Equipment decontamination will be performed at Level D protection described below, unless an upgrade in protection is required during the sampling event. If an upgrade is required, decontamination will be performed at that level.

- **SAMPLING**
  Sediment, surface water, and fish-tissue sampling from a boat will be performed using U.S. Coast Guard-approved personal floatation devices with Level D protection and latex or nitrile gloves. The boat will have sufficient capacity and stability to support the samplers and equipment.

  Sampling from the shore or in shallow water (less than two feet) will be done with boots or waders in addition to the Level D protection listed below.

- **MISCELLANEOUS ACTIVITIES**
  RI field activities not listed above will be done using Level D protective equipment. The use of gloves will be based on whether potential contaminated material/equipment is being handled (i.e., video camera).

Level D protection includes the following clothing and hand protection:

- Work clothes,
- Latex or nitrile gloves,
- Safety work books/shoes, and
- Safety glasses.

A Tyvek coverall will be worn when potentially impacted material is being handled. An upgrade in protection to Level C will include the following respiratory and dermal protection:

- Full-face, air-purifying respirator with GMC-H organic vapor/acid gas high-efficiency particulate filter cartridge,
- Latex inner gloves,
- Safety work books/shoes,
- Nitrile outer gloves, and
- Liquid-resistant Tyvek coverall.

Level C protection is not anticipated based upon RI tasks and historical sample results.

5.2 MODIFICATION FOR PERSONAL PROTECTION REQUIREMENTS

Modifications will be made as conditions warrant. These modifications will be documented and approved by the Project HSC.

6.0 MONITORING EQUIPMENT

A photoionization detector (PID) (HNu with 10.2 eV probe or equivalent) will be used during sampling activities involving the handling of potentially impacted aqueous media (i.e., landfill seeps) and sediment. This equipment is used to provide warning and allow for appropriate action to be taken to prevent exposure from contaminants released into the atmosphere. Use of the PID during surveying tasks is optional and will be determined by the HSR.

An oxygen/explosimeter (i.e., O_2/LEL meter) will be used if entry into storm sewers is required to collect samples. The O_2/LEL of the storm sewer atmosphere will be monitored initially to determine if permitted confined space entry procedures are required. If the atmosphere is acceptable to enter, air monitoring will continue during sampling until field personnel exit the sewer.

6.1 ACTION LEVELS FOR DETERMINING PROTECTION LEVELS

The level of protection will be determined according to sustained concentrations of vapors detected with the PID. If, at any time, sustained VOC concentrations exceed background levels in the workers' breathing zone during Level D activities, an upgrade to Level C will be warranted. If sustained VOC concentrations exceed 5 parts per million (ppm) above background levels in the workers' breathing zone for Level C activities, an upgrade to Level B will be warranted.

For the purpose of this HASP, breathing zone is defined as that zone above the worker's waist. Background level is defined as the concentration of VOCs in an area free of site-generated airborne contaminants (generally located upwind of the work area). Instruments will be calibrated and operated in accordance with manufacturer's specifications.

Background will be determined on a daily basis. Based on the measurements and the activities being performed, the Project HSC will determine the protection level and procedures to be followed upon continuation of the work.

If O_2/LEL readings in the sewer exceed 5 percent LEL or O_2 concentration fall outside the range of 19.5 percent to 21 percent, field personnel will immediately evacuate the sewer and notify the HSR. Other options for sample collection will be evaluated and discussed with the HSC (e.g., SCBAs, ventilation, etc.).

6.2 HEALTH AND SAFETY EQUIPMENT CALIBRATION

The air monitoring equipment will be calibrated daily prior to initiating on-site work activities. Calibration records will be entered on the daily calibration record form (Attachment C-2) and maintained separately.

7.0 STANDARD OPERATING PROCEDURES

The following standard operating procedures (SOPs) will be adhered to by all Cummings/Riter personnel for on-site activities related to this project:

- No surface water samples will be collected during high-flow events. Determination of high-flow events will be made by the HSR.
- The "buddy system" will be used during the performance of potentially dangerous activities including activities involving a boat. Operation of the boat will only be during daylight hours.
- U.S. Coast Guard-approved personal floatation devices are to be worn when working in water deeper than two feet. This includes standing in water.
- Eating, drinking, chewing gum or tobacco, smoking, or any other practice which increases the potential for hand-to-mouth transfer and ingestion of material is strictly prohibited during sampling activities. Areas will be designated for such activities.
- Potential contamination avoidance should be practiced. Wherever possible, personnel should not walk or sit in potentially contaminated areas.
- Cummings/Riter, AMEC, and other field personnel must adhere to the information contained in this HASP.
- A safety meeting is mandatory before initiating work and periodically thereafter, as needed.
- For Cummings/Riter and AMEC personnel, this HASP incorporates, by reference, the policies and procedures established by their respective Corporate Health and Safety Programs.

7.1 SITE ENTRY PROCEDURES

Prior to performance of on-site activities related to this HASP, the following procedures will be performed:

- The HSR will review the contents of this HASP with project personnel who will be on site, and answer any questions regarding its content.
- The air monitoring equipment will be checked and calibrated.
- Personnel will dress out in the appropriate level of protection at the work area.

The number of RI workers on site at Koppers Pond is anticipated to be less than six at a time.

7.2 SITE EXIT PROCEDURES

Prior to leaving the site, the following procedures will be performed:

- Personnel shall undergo personal decontamination,
- Personnel shall ensure that the work area and equipment are secured, and
- Disposable clothing and equipment will be placed in plastic bags for proper disposal.

7.3 HEAT-TRESS MONITORING

Heat stress is not a major concern because extensive protective equipment will not be required for most activities. Heat-stress monitoring of personnel wearing impervious clothing shall commence when the ambient temperature is 70 degrees Fahrenheit (°F) or above. Frequency of monitoring shall increase as the ambient temperature rises. Various control measures shall be employed if heat stress becomes a problem. These include the following:

- Provision for liquids to replace body fluids,
- Establishment of a work regimen that allows for rest periods to cool down, and
- Training of workers in the prevention of heat stress.

7.4 BIOLOGICAL HAZARDS

Biological hazards, including snakes, poisonous vegetation, and insects, are likely to be encountered during the RI activities. The use of PPE, including long-sleeved shirts and work boots in heavy vegetation areas will minimize exposure. Also, decontamination after contact with insects or vegetation will reduce exposure symptoms. The use of

insect repellent will aid in controlling biological hazards due to insects. When working in high tick areas, disposable Tyvek coveralls and a hat can be worn. Leg and arm openings can be taped to boots and gloves.

8.0 DECONTAMINATION

Personnel working at the project site may become impacted in a number of ways, including the following:

- Contacting vapors, gases, mists, or particulates in the air,
- Being splashed by impacted materials,
- Walking through puddles or liquids or on impacted sediments, and
- Using impacted instruments or equipment.

The use of protective clothing and respirators help prevent the wearer from becoming impacted or inhaling constituents, while good work practices help reduce the contamination of protective clothing, instruments, and equipment.

Even with these safeguards, the risk of potential exposure remains. Harmful materials can be transferred into clean areas, exposing unprotected personnel. To prevent such occurrences, decontamination procedures have been developed and will be implemented.

The extent of required decontamination measures depends on the following factors:

- Type of contaminants,
- Amount and concentration of contamination,
- Levels of protection worn,
- Reason for leaving impacted zone, and
- Work function.

The wide variation of site activities and exposure potential does not allow for the use of one general constituent reduction procedure; instead, several procedures will be used depending on the activity. These procedures are described in the following subsections.

8.1 SMALL TOOLS

Small tools and other apparatus used for sampling, such as trowels, spoons, corers, or borers, will be washed in a detergent and water solution (e.g., Alconox® or Liquinox®) and rinsed with tap water to remove particulates. Field filtration equipment (if required)

will be rinsed with dilute nitric acid. The equipment will then be rinsed with methanol. The final step will be a distilled or deionized water rinse. Following decontamination, the equipment will be wrapped in aluminum foil to prevent possible contamination prior to the next use.

A similar decontamination protocol will be employed by the analytical laboratory when the fish are being prepared for fillet samples or homogenates.

8.2 MONITORING EQUIPMENT

Monitoring equipment, including water level sensors, pH probes, slugs, and pressure transducers, will be rinsed with distilled water and methanol between uses.

8.3 VEHICLES

Any vehicle working in the Exclusion Zone will be cleaned with a high-pressure, hot-water spray before leaving the site. Each vehicle will be inspected after cleaning for any soil or sludge remaining on the tires or elsewhere by the HSR. Vehicles that were used in the Exclusion Zone will be cleaned to the satisfaction of the HSR or his designated assistant prior to leaving the site.

8.4 PERSONNEL

The project area will have an area for the workers to don, store, and remove protective equipment. Prior to removal of protective equipment, personnel will remove constituents from boots, gloves, and disposable suits in the Contamination Reduction Zone. A soap wash followed by a water rinse will be sufficient in most cases.

Disposable coveralls and gloves will be placed in plastic bags for disposal. If other protective equipment is thoroughly impacted, the HSR may decide to dispose of this equipment rather than to try to clean the equipment.

Personnel will wash hands and face following removal of protective clothing or contact with vegetation. Personnel wash-water residues will be collected, and properly disposed of.

8.5 INVESTIGATION-DERIVED WASTE

With the permission of CBS Corporation (CBS), the liquid investigation-derived waste (IDW) will be disposed of at the barrier well groundwater treatment plant located at the former Westinghouse Horseheads plant site. Characterization of any such liquid IDW will be in accordance with CBS directions prior to disposal.

Solid IDW from field sampling activities will be disposed of as commercial trash. Excess samples, including both abiotic and fish samples, will be disposed of by the laboratory in accordance with their SOPs and any applicable permit requirements.

9.0 SITE-SPECIFIC TRAINING

The Project HSC will implement a site-specific training program for project employees. The HSR, as the on-site representative of the Project HSC, will instruct employees in proper material handling techniques; proper methods for the use, storage, and disposal of decontamination fluids; preventive maintenance of safety equipment; personal hygiene practices; and proper use of PPE.

The training program will provide instruction for site employees on responding effectively to an emergency. The appropriate response to fire, explosions, and the shutdown of operations will be reviewed. Project employees will be instructed as to the proper response to field monitoring results. Emergency procedures, areas of the site that have restricted access, methods used for project decontamination, and general safety will also be covered in the training.

The project-specific training program will cover the following topics:

- Site history,
- Project organization,
- Explanation of effects of toxic chemicals identified at the site,
- Explanation of biological and physical hazards,
- Requirements of personal protection (e.g., respirators, etc.),
- Prohibited actions or procedures,
- Safety precautions,
- Safe operation of boat used for RI activities,
- Emergency procedures,
- Decontamination procedures,
- Work area, and
- Air monitoring program.

Prior to working on site, replacement employees will be required to receive the initial training given by the HSR. Records of personnel attendance at training sessions will be maintained on site.

Safety meetings will be held by the HSR to discuss safety problems, changes in site conditions, monitoring results, or other safety related topics. Attendance lists, including signatures and topics discussed, for safety meetings will be maintained on site.

10.0 REPORTS AND RECORD KEEPING

Records of health and safety activities for the RI will be maintained by Cummings/Riter and other firms. The records will document air monitoring levels, exposure levels, protective equipment worn, incidents, medical monitoring, and training.

10.1 LOGS AND REPORTS

The HSR shall maintain logs and reports covering the implementation of this HASP. Typical logs/reports include the following:

- **TRAINING LOGS (SHALL BE COMPLETED FOR BOTH INITIAL TRAINING AND REFRESHER TRAINING)**
  - Employee signatures,
  - Topics covered,
  - Materials used,
  - Equipment demonstration,
  - Equipment practice for each employee,
  - Date, and
  - Time.

- **DAILY LOGS**
  - Date,
  - Area (site-specific) checks,
  - Equipment utilized by employees and job function,
  - Protective clothing and devices worn by employees,
  - Violations of the HASP,
  - Instances of job-related injuries and illness,
  - Area monitoring results, and
  - HSR signature and date.

- **INCIDENT REPORT**
  Describes injuries, off-site release, or accident (will be reported in writing to the project manager and the Group Representative within 48 hours of incident).

- **MEDICAL CERTIFICATIONS**
  Reviewed by Project HSC prior to employee working on site.

CUMMINGS RITER

10.2 RECORD KEEPING

Cummings/Riter maintains health and safety records in accordance with OSHA regulations. Access to records by employees is permitted as required under state and federal regulations. Medical files are confidential and access to these files will only be provided to parties allowed by federal law.

11.0 CONTINGENCY PLAN

Emergency response procedures have been developed to cover extraordinary conditions that may occur during the Kentucky Avenue Wellfield Operable Unit 4 RI activities.

11.1 GENERAL RESPONSE CONSIDERATIONS

Emergencies must be dealt with in a manner that minimizes health and safety risks to site personnel and the public. Site personnel will not be required to perform emergency-related tasks for which they have not received training.

The following procedures shall be implemented in the event of an emergency:

- First aid or other appropriate initial action will be administered by those closest to the accident/event. This assistance will be coordinated by the ranking individual on site and will be conducted in a manner so that those rendering assistance are not placed in a situation of unacceptable risk. The primary concern is to avoid placing a greater number of workers in jeopardy.

- Employees shall immediately report all accidents and unusual events to:
  - Project HSC,
  - Project Manager,
  - HSR, and
  - The Group Project Coordinator.

- The HSR is responsible for conducting the emergency response in an efficient, rapid, and safe manner. The HSR will decide if off-site assistance and/or medical treatment are required, and shall be responsible for alerting off-site authorities and arranging for their assistance.

- The HSR will provide to the above-referenced personnel an Incident Report which includes the following:
  - A description of the incident (including date, time, and duration);
  - Date, time, and name of all persons/agencies notified and their response; and
  - A description of corrective actions implemented or other resolution of the incident.

- All workers on site are responsible for conducting themselves in a mature, calm manner in the event of an accident/unusual event to avoid spreading the danger to themselves and to surrounding workers.

11.2 RESPONSIBILITIES

The HSR or a designated substitute shall have responsibility for directing response activities in the event of an emergency. He/she will perform the following:

- Assess the situation;
- Determine required response measures;
- Notify appropriate response teams;
- Determine and direct on-site personnel during the emergency; and
- With the Group Project Coordinator, contact and coordinate with government agencies.

The HSR or a designated substitute shall coordinate response activities with those of public agencies as follows:

• IMMEDIATE EMERGENCIES	PHONE NUMBERS
Police, Fire and Ambulance	911
• EMERGENCY SUPPORT	
Group Project Coordinator (Mr. Leo Brausch)	(724) 444-0377
Cummings/Riter Office	(412) 241-4500
Hardinge Brothers	(607) 734-2281
Qualisys (medical consultants)	(800) 874-4676
USEPA (24-hour hotline)	(800) 424-8802
USEPA (Ms. Isabel Rodrigues)	(212) 637-4248
New York State Department of Environmental Conservation (NYSDEC) Region 8	(716) 226-2466
AMEC Westford Office (Dr. Paul Anderson)	(978) 692-9090
AMEC-Portland Office (Dr. Russ Keenan)	(207) 879-4222

Cummings/Riter project staff will have available the home phone numbers for their HSR, Project HSC, and project manager. No work is expected to be done during non-daylight hours or on weekends.

11.3 EMERGENCY RESPONSE EQUIPMENT

Before site operations are initiated, the following emergency equipment will be provided at the site:

- Portable eyewash stations (hand held),
- Cellular telephone (HSR),
- List of persons and phone numbers for emergency notification, and
- Locations of water for washing hands and face.

There are restroom facilities inside the water treatment building on the former Westinghouse facility property.

11.4 SITE EMERGENCY

Procedures for emergency evacuation will be established for the work area even though the contaminants being handled and the procedures employed make this an extremely unlikely occurrence. The rendezvous point will be the entrance to the Hardinge Brothers facility.

11.5 HOSPITAL

St. Joseph's Hospital is the identified hospital for the Kentucky Avenue Wellfield Superfund Site. A map to St. Joseph's Hospital with driving directions is included as Attachment C-3. The emergency procedures provide for the use of an ambulance service to take injured personnel to the hospital. This will be the procedure for this project. The following information shall be given for directions to the emergency agency:

- **KOPPERS POND OR DOWNSTREAM INCIDENTS:**
  Hardinge Brothers
  1 Hardinge Drive
  Horseheads, New York

- **INDUSTRIAL DRAINAGEWAY INCIDENTS:**
  Young's Tire Store
  909 Chemung Street
  Village of Horseheads
  Horseheads, New York

CUMMINGS RITER

ATTACHMENT C-1

MSDSs FOR ALCONOX, NITRIC ACID, METHANOL, AND HEXANE

ALCONOX MSDS

Section 1 : MANUFACTURER INFORMATION

Product name: Alconox

Supplier: Same as manufacturer.

Manufacturer: Alconox, Inc.
30 Glenn St.
Suite 309
White Plains, NY 10603.

Manufacturer emergency phone number: 800-255-3924.
813-248-0585 (outside of the United States).

Manufacturer: Alconox, Inc.
30 Glenn St.
Suite 309
White Plains, NY 10603.

Supplier MSDS date: 2005/03/09

D.O.T. Classification: Not regulated.

Section 2 : HAZARDOUS INGREDIENTS

C.A.S.	CONCENTRATION %	Ingredient Name	T.L.V.	LD/50	LC/50
25155-30-0	10-30	SODIUM DODECYLBENZENESULFONATE	NOT AVAILABLE	438 MG/KG RAT ORAL 1330 MG/KG MOUSE ORAL	NOT AVAILABLE
497-19-8	7-13	SODIUM CARBONATE	NOT AVAILABLE	4090 MG/KG RAT ORAL 6600 MG/KG MOUSE ORAL	2300 MG/M3/2H RAT INHALATION 1200 MG/M3/2H MOUSE INHALATION
7722-88-5	10-30	TETRASODIUM PYROPHOSPHATE	5 MG/M3	4000 MG/KG RAT ORAL 2980 MG/KG MOUSE ORAL	NOT AVAILABLE
7758-29-4	10-30	SODIUM PHOSPHATE	NOT AVAILABLE	3120 MG/KG RAT ORAL 3100 MG/KG MOUSE ORAL >4640 MG/KG RABBIT DERMAL	NOT AVAILABLE

Section 2A : ADDITIONAL INGREDIENT INFORMATION

Note: (supplier).
CAS# 497-19-8: LD50 4020 mg/kg - rat oral.
CAS# 7758-29-4: LD50 3100 mg/kg - rat oral.

Section 3 : PHYSICAL / CHEMICAL CHARACTERISTICS

Physical state: Solid

Appearance & odor: Almost odourless.
White granular powder.

Odor threshold (ppm): Not available.

Vapour pressure (mmHg): Not applicable.

Vapour density (air=1): Not applicable.

By weight: Not available.

Evaporation rate (butyl acetate = 1): Not applicable.

Boiling point (°C): Not applicable.

Freezing point (°C): Not applicable.

pH: (1% aqueous solution).
9.5

Specific gravity @ 20 °C: (water = 1).
0.85 - 1.10

Solubility in water (%): 100 - > 10% w/w

Coefficient of water\oil dist.: Not available.

VOC: None

Section 4 : FIRE AND EXPLOSION HAZARD DATA

Flammability: Not flammable.

Conditions of flammability: Surrounding fire.

Extinguishing media: Carbon dioxide, dry chemical, foam.
Water
Water fog.

Special procedures: Self-contained breathing apparatus required.
Firefighters should wear the usual protective gear.

Auto-ignition temperature: Not available.

Flash point (°C), method: None

Lower flammability limit (% vol): Not applicable.

Upper flammability limit (% vol): Not applicable.

Not available.

Sensitivity to mechanical impact: Not applicable.

Hazardous combustion products: Oxides of carbon (COx).
Hydrocarbons.

Rate of burning: Not available.

Explosive power: None

Section 5 : REACTIVITY DATA

Chemical stability: Stable under normal conditions.

Conditions of instability: None known.

Hazardous polymerization: Will not occur.

Incompatible substances: Strong acids.
Strong oxidizers.

Hazardous decomposition products: See hazardous combustion products.

Section 6 : HEALTH HAZARD DATA

Route of entry: Skin contact, eye contact, inhalation and ingestion.

Effects of Acute Exposure

Eye contact: May cause irritation.

Skin contact: Prolonged contact may cause irritation.

Inhalation: Airborne particles may cause irritation.

Ingestion: May cause vomiting and diarrhea.
May cause abdominal pain.
May cause gastric distress.

Effects of chronic exposure: Contains an ingredient which may be corrosive.

LD50 of product, species & route: > 5000 mg/kg rat oral.

LC50 of product, species & route: Not available for mixture, see the ingredients section.

Exposure limit of material: Not available for mixture, see the ingredients section.

Sensitization to product: Not available.

Carcinogenic effects: Not listed as a carcinogen.

Reproductive effects: Not available.

Teratogenicity: Not available.

Mutagenicity: Not available.

Synergistic materials: Not available.

Medical conditions aggravated by exposure: Not available.

First Aid

Skin contact: Remove contaminated clothing.
Wash thoroughly with soap and water.
Seek medical attention if irritation persists.

Eye contact: Check for and remove contact lenses.
Flush eyes with clear, running water for 15 minutes while holding eyelids open: if irritation persists, consult a physician.

Inhalation: Remove victim to fresh air.
Seek medical attention if symptoms persist.

Ingestion: Dilute with two glasses of water.
Never give anything by mouth to an unconscious person.
Do not induce vomiting, seek immediate medical attention.

Section 7 : PRECAUTIONS FOR SAFE HANDLING AND USE

Leak/Spill: Contain the spill.
Recover uncontaminated material for re-use.
Wear appropriate protective equipment.
Contaminated material should be swept or shoveled into appropriate waste container for disposal.

Waste disposal: In accordance with municipal, provincial and federal regulations.

Handling procedures and equipment: Protect against physical damage.
Avoid breathing dust.
Wash thoroughly after handling.
Keep out of reach of children.
Avoid contact with skin, eyes and clothing.
Launder contaminated clothing prior to reuse.

Storage requirements: Keep containers closed when not in use.
Store away from strong acids or oxidizers.
Store in a cool, dry and well ventilated area.

Section 8 : CONTROL MEASURES

Precautionary Measures

Gloves/Type:

Neoprene or rubber gloves.

Respiratory/Type:

If exposure limit is exceeded, wear a NIOSH approved respirator.

Eye/Type:

Safety glasses with side-shields.

Footwear/Type: Safety shoes per local regulations.

Clothing/Type: As required to prevent skin contact.

Other/Type: Eye wash facility should be in close proximity.
Emergency shower should be in close proximity.

Ventilation requirements: Local exhaust at points of emission.

MSDS Number: **H2381** * * * * * *Effective Date: **05/07/07** * * * * * *Supercedes: **08/10/04***

MSDS Material Safety Data Sheet

From: Mallinckrodt Baker, Inc.
222 Red School Lane
Phillipsburg, NJ 08865

Mallinckrodt CHEMICALS J.T.Baker

24 Hour Emergency Telephone: 908-859-2151
CHEMTREC: 1-800-424-9300

National Response in Canada
CANUTEC: 613-996-6666

Outside U.S. and Canada
Chemtrec: 703-527-3887

NOTE: CHEMTREC, CANUTEC and National Response Center emergency numbers to be used only in the event of chemical emergencies involving a spill, leak, fire, exposure or accident involving chemicals

All non-emergency questions should be directed to Customer Service (1-800-582-2537) for assistance

HEXANE

1. Product Identification

Synonyms: Hexanes,Normal Hexane; Hexyl Hydride; Hexane 95%
CAS No.: 110-54-3 (n-hexane)
Molecular Weight: 86.18
Chemical Formula: CH3(CH2)4CH3 n-hexane
Product Codes:
J.T. Baker: 9262, 9304, 9308, N168
Mallinckrodt: 5186

2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Hexane	110-54-3	85 - 100%	Yes
Methylcyclopentane	96-37-7	1 - 2%	Yes
Trace amount of Benzene (10 ppm)	071-43-2	*	No

3. Hazards Identification

Emergency Overview

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.

J.T. Baker SAF-T-DATA(tm) Ratings (Provided here for your convenience)

Health Rating: 2 - Moderate
Flammability Rating: 3 - Severe (Flammable)
Reactivity Rating: 0 - None

Contact Rating: 2 - Moderate
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER
Storage Color Code: Red (Flammable)

Potential Health Effects

The health hazards addressed are for the major component: n-hexane.

Inhalation:
Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Greater exposure may cause muscle weakness, numbness of the extremities, unconsciousness and death.
Ingestion:
May produce abdominal pain, nausea. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms expected to parallel inhalation.
Skin Contact:
May cause redness, irritation, with dryness, cracking.
Eye Contact:
Vapors may cause irritation. Splashes may cause redness and pain.
Chronic Exposure:
Repeated or prolonged skin contact may defat the skin and produce irritation and dermatitis. Chronic inhalation may cause peripheral nerve disorders and central nervous system effects.
Aggravation of Pre-existing Conditions:
Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance. May affect the developing fetus.

4. First Aid Measures

Inhalation:
Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:
Aspiration hazard. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention immediately.
Skin Contact:
Remove any contaminated clothing. Wipe off excess from skin. Wash skin with soap and water for at least 15 minutes. Get medical attention if irritation develops or persists.
Eye Contact:
Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

Note to Physician:
BEI=2,5-hexadione in urine, sample at end of shift at workweeks end, 5 mg/g creatine. Also, measure n-hexane in expired air. Analgesics may be necessary for pain management, there is no specific antidote. Monitor arterial blood gases in cases of severe aspiration.

5. Fire Fighting Measures

Fire:
Flash point: -23C (-9F) CC
Autoignition temperature: 224C (435F)
Flammable limits in air % by volume:

lel: 1.2; uel: 7.7
Extremely Flammable Liquid and Vapor! Vapor may cause flash fire. Dangerous fire hazard when exposed to heat or flame.

Explosion:
Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Contact with oxidizing materials may cause extremely violent combustion.Explodes when mixed @ 28C with dinitrogen tetraoxide. Sensitive to static discharge.

Fire Extinguishing Media:
Dry chemical, foam or carbon dioxide. Water may be ineffective.

Special Information:
In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Water spray may be used to keep fire exposed containers cool. Vapors can flow along surfaces to distant ignition source and flash back. Vapor explosion hazard exists indoors, outdoors, or in sewers.

6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB® solvent adsorbent is recommended for spills of this product.

7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from direct sunlight and any area where the fire hazard may be acute. Store in tightly closed containers (preferably under nitrogen atmosphere). Outside or detached storage is preferred. Inside storage should be in a standard flammable liquids storage room or cabinet. Separate from oxidizing materials. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

8. Exposure Controls/Personal Protection

Airborne Exposure Limits:
N-Hexane [110-54-3]:
-OSHA Permissible Exposure Limit (PEL): 500 ppm (TWA)
-ACGIH Threshold Limit Value (TLV): 50 ppm (TWA), Skin
other isomers of hexane
-ACGIH Threshold Limit Value (TLV): 500 ppm (TWA),1000ppm (STEL)

Ventilation System:
A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

Personal Respirators (NIOSH Approved):
If the exposure limit is exceeded and engineering controls are not feasible, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Breathing air quality must meet the requirements of the OSHA respiratory protection standard (29CFR1910.134).
Skin Protection:
Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.
Eye Protection:
Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

9. Physical and Chemical Properties

Appearance:
Clear, colorless liquid.
Odor:
Light odor.
Solubility:
Insoluble in water.
Specific Gravity:
0.66
pH:
No information found.
% Volatiles by volume @ 21C (70F):
100
Boiling Point:
ca. 68C (ca. 154F)
Melting Point:
ca. -95C (ca. -139F)
Vapor Density (Air=1):
3.0
Vapor Pressure (mm Hg):
130 @ 20C (68F)
Evaporation Rate (BuAc=1):
9

10. Stability and Reactivity

Stability:
Stable under ordinary conditions of use and storage. Heat will contribute to instability.
Hazardous Decomposition Products:
May produce acrid smoke and irritating fumes when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizers.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

11. Toxicological Information

N-Hexane: Oral rat LD50: 28710 mg/kg. Irritation eye rabbit: 10 mg mild. Investigated as a tumorigen, mutagen

and reproductive effector.

--------\Cancer Lists\----------

Ingredient	---NTP Carcinogen--- Known	Anticipated	IARC Category
Hexane (110-54-3)	No	No	None
Methylcyclopentane (96-37-7)	No	No	None
Trace amount of Benzene (10 ppm) (071-43-2)	Yes	No	1

12. Ecological Information

Environmental Fate:
When released into the soil, this material may biodegrade to a moderate extent. When released into the soil, this material is not expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material may biodegrade to a moderate extent. When released to water, this material is expected to quickly evaporate. When released into the water, this material is expected to have a half-life between 1 and 10 days. This material has an estimated bioconcentration factor (BCF) of less than 100. This material has a log octanol-water partition coefficient of greater than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material is expected to be readily degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material is expected to have a half-life between 1 and 10 days.
Environmental Toxicity:
No information found.

13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Domestic (Land, D.O.T.)

Proper Shipping Name: HEXANES
Hazard Class: 3
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

International (Water, I.M.O.)

Proper Shipping Name: HEXANES
Hazard Class: 3
UN/NA: UN1208
Packing Group: II
Information reported for product/size: 215L

15. Regulatory Information

--------\Chemical Inventory Status - Part 1\----------

Ingredient	TSCA	EC	Japan	Australia
Hexane (110-54-3)	Yes	Yes	Yes	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
Trace amount of Benzene (10 ppm) (071-43-2)	Yes	Yes	Yes	Yes

--------\Chemical Inventory Status - Part 2\----------

Ingredient	Korea	--Canada-- DSL	NDSL	Phil.
Hexane (110-54-3)	Yes	Yes	No	Yes
Methylcyclopentane (96-37-7)	Yes	Yes	No	Yes
Trace amount of Benzene (10 ppm) (071-43-2)	Yes	Yes	No	Yes

--------\Federal, State & International Regulations - Part 1\----------

Ingredient	-SARA 302- RQ	TPQ	------SARA 313------ List	Chemical Catg.
Hexane (110-54-3)	No	No	Yes	No
Methylcyclopentane (96-37-7)	No	No	No	No
Trace amount of Benzene (10 ppm) (071-43-2)	No	No	Yes	No

--------\Federal, State & International Regulations - Part 2\----------

Ingredient	CERCLA	-RCRA- 261.33	-TSCA- 8(d)
Hexane (110-54-3)	5000	No	No
Methylcyclopentane (96-37-7)	No	No	No
Trace amount of Benzene (10 ppm) (071-43-2)	10	U019	No

Chemical Weapons Convention: No TSCA 12(b): No CDTA: No
SARA 311/312: Acute: Yes Chronic: Yes Fire: Yes Pressure: No
Reactivity: No (Mixture / Liquid)

WARNING:
THIS PRODUCT CONTAINS A CHEMICAL(S) KNOWN TO THE STATE OF CALIFORNIA TO CAUSE CANCER.

Australian Hazchem Code: 3[Y]E
Poison Schedule: None allocated.
WHMIS:
This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

16. Other Information

NFPA Ratings: Health: **1** Flammability: **3** Reactivity: **0**
Label Hazard Warning:
DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.
Label Precautions:
Keep away from heat, sparks and flame.
Keep container closed.
Use only with adequate ventilation.
Wash thoroughly after handling.
Avoid breathing vapor or mist.
Avoid contact with eyes, skin and clothing.
Label First Aid:
Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person.

Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. In all cases call a physician.

Product Use:
Laboratory Reagent.

Revision Information:
No Changes.

Disclaimer:
****

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Prepared by: Environmental Health & Safety
Phone Number: (314) 654-1600 (U.S.A.)

NITRIC ACID

0183
April 1994

CAS No: 7697-37-2
RTECS No: QU5775000
UN No: 2031
EC No: 007-004-00-1

Concentrated Nitric Acid (70%)
HNO_3
Molecular mass: 63.0

TYPES OF HAZARD/ EXPOSURE	ACUTE HAZARDS/SYMPTOMS	PREVENTION	FIRST AID/FIRE FIGHTING
FIRE	Not combustible but enhances combustion of other substances. Gives off irritating or toxic fumes (or gases) in a fire.	NO contact with flammable substances. NO contact with combustibles or organic chemicals.	In case of fire in the surroundings: NO foam.
EXPLOSION	Risk of fire and explosion on contact with many common organic compounds.		In case of fire: keep drums, etc., cool by spraying with water.
EXPOSURE		**AVOID ALL CONTACT!**	
Inhalation	Burning sensation. Cough. Laboured breathing. Unconsciousness. Symptoms may be delayed (see Notes).	Ventilation, local exhaust, or breathing protection.	Fresh air, rest. Half-upright position. Artificial respiration may be needed. Refer for medical attention.
Skin	Corrosive. Serious skin burns. Pain. Yellow discolouration.	Protective clothing.	Remove contaminated clothes. Rinse skin with plenty of water or shower. Refer for medical attention.
Eyes	Corrosive. Redness. Pain. Severe deep burns.	Face shield or eye protection in combination with breathing protection.	First rinse with plenty of water for several minutes (remove contact lenses if easily possible), then take to a doctor.
Ingestion	Corrosive. Abdominal pain. Burning sensation. Shock.	Do not eat, drink, or smoke during work. Wash hands before eating.	Do NOT induce vomiting. Give plenty of water to drink. Rest. Refer for medical attention.

SPILLAGE DISPOSAL	PACKAGING & LABELLING	
Evacuate danger area! Consult an expert! Ventilation. Collect leaking liquid in sealable containers. Cautiously neutralize remainder with sodium carbonate. Then wash away with plenty of water. Do NOT absorb in saw-dust or other combustible absorbents. Personal protection: complete protective clothing including self-contained breathing apparatus.	O Symbol C Symbol R: 8-35 S: (1/2-)23-26-36-45 Note: B UN Hazard Class: 8 UN Pack Group: II	Unbreakable packaging; put breakable packaging into closed unbreakable container. Do not transport with food and feedstuffs.

EMERGENCY RESPONSE	SAFE STORAGE
Transport Emergency Card: TEC (R)-80S2031-II or 80GO1-I NFPA Code: H 3; F 0; R 0; OX	Separated from combustible and reducing substances, bases, food and feedstuffs, organic chemicals. Cool. Dry. Keep in a well-ventilated room.

IPCS
International Programme on Chemical Safety

Prepared in the context of cooperation between the International Programme on Chemical Safety and the European Commission
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SEE IMPORTANT INFORMATION ON THE BACK.

IMPORTANT DATA

Physical State; Appearance
COLOURLESS TO YELLOW LIQUID, WITH PUNGENT ODOUR.

Chemical dangers
The substance decomposes on warming producing nitrogen oxides. The substance is a strong oxidant and reacts violently with combustible and reducing materials, e.g., turpentine, charcoal, alcohol. The substance is a strong acid, it reacts violently with bases and is corrosive to metals. Reacts very violently with organic chemicals (e.g., acetone, acetic acid, acetic anhydride), causing fire and explosion hazard. Attacks some plastics.

Occupational exposure limits
TLV: 2 ppm as TWA, 4 ppm as STEL; (ACGIH 2004).
MAK: 2 ppm, 5.2 mg/m^3; Peak limitation category: I(1); Pregnancy risk group: IIc; (DFG 2004).

Routes of exposure
The substance can be absorbed into the body by inhalation of its vapour and by ingestion.

Inhalation risk
A harmful contamination of the air can be reached very quickly on evaporation of this substance at 20•C.

Effects of short-term exposure
The substance is very corrosive to the eyes, the skin and the respiratory tract. Corrosive on ingestion as well. Inhalation of vapour may cause lung oedema (see Notes).

PHYSICAL PROPERTIES

Boiling point: 121•C
Melting point: -41.6•C
Relative density (water = 1): 1.4
Solubility in water: miscible

Vapour pressure, kPa at 20•C: 6.4
Relative vapour density (air = 1): 2.2
Relative density of the vapour/air-mixture at 20•C (air = 1): 1.07

ENVIRONMENTAL DATA

NOTES

Depending on the degree of exposure, periodic medical examination is suggested.
The symptoms of lung oedema often do not become manifest until a few hours have passed and they are aggravated by physical effort. Rest and medical observation are therefore essential.
Rinse contaminated clothes (fire hazard) with plenty of water.
Other UN 2031 classification with more than 70% nitric acid, hazard class 8, subsidiary hazard 5.1, packing group I.
Card has been partly updated in April 2005. See sections Occupational Exposure Limits, Emergency Response, Notes.

ADDITIONAL INFORMATION

LEGAL NOTICE | Neither the EC nor the IPCS nor any person acting on behalf of the EC or the IPCS is responsible

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Schenectady, NY 12303-1836 USA
(518) 377-8854

Material Safety Data Sheets Collection:

Sheet No. 354
Methyl Alcohol

Issued: 11/77 Revision: D, 11/91

Section 1. Material Identification

Methyl alcohol (CH_3OH) Description: Derived from destructive distillation of wood, oxidation of hydrocarbons, or high-pressure catalytic synthesis from hydrogen and carbon dioxide or carbon monoxide. Used as a solvent in manufacturing industrial chemicals and chemical pharmaceuticals, a raw material for making formaldehyde and methyl esters, a softening agent for pyroxylin plastics, a dehydrator for natural gas, a feedstock for manufacturing synthetic proteins by continuous fermentation, an octane booster in gasoline, an extractant for animal and vegetable oils; in antifreeze for automotive radiators, air brakes, gasoline, and diesel oil; and in denaturing ethanol.
Other Designations: CAS No. 67-56-1, carbinol, Columbian spirits, methanol, methyl hydroxide, methylol, monohydroxymethane, pyroxylic spirit, wood alcohol, wood naphtha, wood spirit.
Manufacturer: Contact your supplier or distributor. Consult latest *Chemical Week Buyers' Guide*(73) for a suppliers list.

R 1
I 2
S 1*
K 4
* Skin absorption

NFPA
3
1 0
-

HMIS
H 2
F 3
R 0
PPG†
† Sec. 8

Cautions: Methyl alcohol is moderately toxic by ingestion and mildly toxic by inhalation and skin absorption. It is flammable, volatile, and a dangerous fire hazard.

Section 2. Ingredients and Occupational Exposure Limits

Methyl alcohol, ca 100%

1990 OSHA PELs (Skin)
8-hr TWA: 200 ppm (260 mg/m³)
15-min STEL: 250 ppm (310 mg/m³)

1990 IDLH Level
25,000 ppm

1991-92 ACGIH TLVs (Skin)
TWA: 200 ppm (262 mg/m³)
STEL: 250 ppm (328 mg/m³)

1990 DFG (Germany) MAK
200 ppm (260 mg/m³)

1990 NIOSH RELs (Skin)
TWA: 200 ppm (260 mg/m³)
Ceiling: 250 ppm (325 mg/m³)

1985-86 Toxicity Data*
Human, inhalation, TC_{Lo}: 300 ppm caused eye (visual field change), CNS (headache), and pulmonary effects
Human, oral, LD_{Lo}: 428 mg/kg causes CNS (headache) and pulmonary (respiratory change) effects
Rat, oral, TD_{Lo}: 7500 mg/kg administered continuously to the female during the 17th to 19th day of gestation produced behavioral effects on newborns
Rat, inhalation, TC_{Lo}: 20,000 ppm/7 hr administered continuously to the female during the 1st to 22nd day of gestation produced specific developmental abnormalities

* See NIOSH, *RTECS* (PC1400000), for additional toxicity data.

Section 3. Physical Data

Boiling Point: 148 °F (64.5 °C)
Freezing Point: -144.04 °F (-97.8 °C)
Vapor Pressure: 29 mm Hg at 68 °F (20 °C)
Vapor Density (air = 1): 1.11
Viscosity: 0.00593 P at 68 °F (20 °C)
Molecular Weight: 32.05
Density: 0.7924 at 68 °F (20 °C)
Water Solubility: Soluble
Other Solubilities: Soluble in ethanol, ether, benzene, ketones, and most organic solvents

Appearance and Odor: Clear, colorless, volatile liquid with a slight alcohol odor when pure, a disagreeably pungent odor when crude, and a low 10-ppm odor threshold.

Section 4. Fire and Explosion Data

Flash Point: 54 °F (12 °C), CC	Autoignition Temperature: 878 °F (470 °C)	LEL: 6% v/v	UEL: 36.5% v/v

Extinguishing Media: For small fires, use dry chemical, carbon dioxide (CO_2), water spray, or alcohol-resistant foam. For large fires, use water spray, fog, or alcohol-resistant foam. Do not scatter material with any more water than needed to extinguish fire.
Unusual Fire or Explosion Hazards: Methyl alcohol is a dangerous fire hazard when exposed to heat, flame, or oxidizers. It is explosive in its vapor form when exposed to heat or flame. Vapors may travel to an ignition source and flash back.
Special Fire-fighting Procedures: Since fire may produce toxic thermal decomposition products, wear a self-contained breathing apparatus (SCBA) with a full facepiece operated in pressure-demand or positive-pressure mode. Also, wear full protective clothing. Structural firefighters' protective clothing is *ineffective* for fires involving methyl alcohol. If possible without risk, remove container from fire area. Apply cooling water to sides of fire-exposed container until fire is well out. Stay away from ends of tanks. Leave area immediately if you hear a rising sound from venting safety device or see any tank discoloration due to fire. Be aware of runoff from fire control methods. Do not release to sewers or waterways.

Section 5. Reactivity Data

Stability/Polymerization: Methyl alcohol is stable at room temperature in closed containers under normal storage and handling conditions. Hazardous polymerization cannot occur.
Chemical Incompatibilities: Methyl alcohol is incompatible with beryllium dihydride, metals (such as potassium or magnesium), oxidants (such as barium perchlorate, bromine, chlorine, hydrogen peroxide, and sodium hypochlorite), potassium tertbutoxide, carbon tetrachloride + metals; reacts explosively with chloroform + heat, and diethyl zinc; and reacts violently with alkyl aluminum salts, acetyl bromide, chloroform + sodium hydroxide, cyanuric chloride, and nitric acid.
Conditions to Avoid: Avoid vapor inhalation and contact with oxidizers and other incompatibles.
Hazardous Products of Decomposition: Thermal oxidative decomposition of methyl alcohol can produce carbon oxides (CO and CO_2), [illegible]le formaldehyde (HCHO) and acrid smoke, and irritating fumes.

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Section 6. Health Hazard Data

Carcinogenicity: In 1990 reports, the IARC, NTP, and OSHA do not list methyl alcohol as a carcinogen.
Summary of Risks: Methyl alcohol is toxic mainly to the nervous system, particularly optic nerves, where damage can progress to permanent blindness. Poisoning may also result in metabolic acidosis. Methyl alcohol oxidizes in the body to form formaldehyde and formic acid. These derivatives are believed responsible for many of methyl alcohol's poisonous and toxic effects. Since it is eliminated slowly from the body, methyl alcohol is considered a cumulative poison. The fatal ingestion dose is 100 to 250 ml, although death is reported from less than 33 ml.
Medical Conditions Aggravated by Long-Term Exposure: None reported
Target Organs: Eyes, central nervous system, skin, and digestive tract.
Primary Entry Routes: Inhalation, ingestion, skin absorption.
Acute effects: Inhalation can cause irritation of eyes and nose, headache, fatigue, nausea, visual impairment (optic nerve neuropathy or visual field changes) or complete and possibly permanent blindness, acidosis, convulsions, circulatory collapse, respiratory failure, and death. Ingestion can cause gastrointestinal (GI) irritation followed by the symptoms described for inhalation and possible kidney impairment. Skin contact results in a feeling of coldness, dryness, and cracking possibly leading to dermatitis. Methyl alcohol can absorb through skin and may cause headache, fatigue, and visual disturbances. Eye contact causes irritation and watering of eyes, inflamed lids, and painful sensitization to light.
Chronic Effects: Chronic inhalation or skin absorption may produce visual impairment or complete blindness.
FIRST AID
Eyes: Gently lift the eyelids and flush immediately and continuously with flooding amounts of water until transported to an emergency medical facility. Do not let victim rub or keep eyes tightly shut. Consult a physician immediately.
Skin: *Quickly* remove contaminated clothing. Since methyl alcohol is volatile and flammable, carefully dispose of contaminated clothing. Rinse with flooding amounts of water for at least 15 min. For reddened or blistered skin, consult a physician. Wash affected area with soap and water.
Inhalation: Remove exposed person to fresh air and support breathing as needed.
Ingestion: Never give anything by mouth to an unconscious or convulsing person. If ingested, have that *conscious and alert* person drink 1 to 2 glasses of water, then induce vomiting.
After first aid, get appropriate in-plant, paramedic, or community medical support.
Note to Physicians: Consider administering 10% ethanol in D5W intravenously to maintain ethyl alcohol blood level at 100 mg/dl. Check formic acid in urine and measure blood pH and plasma bicarbonate. After ingestion, there is typically an 18- to 48-hr latency period before clinical toxicity

Section 7. Spill, Leak, and Disposal Procedures

Spill/Leak: Notify safety personnel, isolate area, deny entry, and stay upwind. Shut off all ignition sources—no flares, smoking, or flames in hazard area. Cleanup personnel should wear fully encapsulating, vapor-protective clothing for spills or leaks with no fire. Water spray may reduce vapor, but not prevent ignition in closed spaces. For small spills, use nonsparking tools to take up with earth, sand, vermiculite, or other absorbent, noncombustible material and place in suitable containers for later disposal. For large spills, dike far ahead of spill and await disposal. Follow applicable OSHA regulations (29 CFR 1910.120).
Environmental Degradation: Aquatic toxicity rating: TLm 96, over 1000 ppm.
Disposal: Contact your supplier or a licensed contractor for detailed recommendations. Follow applicable Federal, state, and local regulations.

EPA Designations
Listed as a RCRA Hazardous Waste (40 CFR 261.33): Hazardous Waste No. U154
CERCLA Hazardous Substance (40 CFR 302.4): Not listed
SARA Extremely Hazardous Substance (40 CFR 355): Not listed
SARA Toxic Chemical (40 CFR 372.65): Not listed

OSHA Designations
Listed as an Air Contaminant (29 CFR 1910.1000, Table Z-1-A)

Section 8. Special Protection Data

Goggles: Wear protective eyeglasses or chemical safety goggles, per OSHA eye- and face-protection regulations (29 CFR 1910.133). Since contact lens use in industry is controversial, establish your own policy.
Respirator: Seek professional advice prior to respirator selection and use. Follow OSHA respirator regulations (29 CFR 1910.134) and, if necessary, wear a NIOSH-approved respirator. Select the respirator based on its suitability to provide adequate worker protection for the given working conditions, level of airborne contamination, and presence of sufficient oxygen. For emergency or nonroutine operations (cleaning spills, reactor vessels, or storage tanks), wear an SCBA. *Warning! Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.*
Other: Wear impervious gloves, boots, aprons, and gauntlets to prevent all skin contact.
Ventilation: Provide general and local explosion-proof exhaust ventilation systems to maintain airborne concentrations below the OSHA PELs (Sec. 2). Local exhaust ventilation is preferred since it prevents contaminant dispersion into the work area by controlling it at its source.[103]
Safety Stations: Make available in the work area emergency eyewash stations, safety/quick-drench showers, and washing facilities.
Contaminated Equipment: Separate contaminated work clothes from street clothes. Launder contaminated work clothing before wearing. Remove this material from your shoes and clean personal protective equipment.
Comments: Never eat, drink, or smoke in work areas. Practice good personal hygiene after using this material, especially before eating, drinking, smoking, using the toilet, or applying cosmetics.

Section 9. Special Precautions and Comments

Storage Requirements: Avoid physical damage to containers. Store in cool, dry, well-ventilated flammables storage area, away from strong oxidizers and other incompatibles. To prevent static sparks, electrically ground all equipment used in methyl alcohol storage, manufacture, and transportation. Use nonsparking tools.
Engineering Controls: To reduce potential health hazards, use sufficient dilution or local exhaust ventilation to control hazardous airborne contaminants and to maintain concentrations at the lowest practical level.
Other Precautions: Consider preplacement and periodic medical examinations of exposed workers emphasizing neurological, kidney, liver, and visual function. Practice good personal hygiene and housekeeping procedures. If respirators are used, institute a respiratory protection program that includes regular training, maintenance, inspection, and evaluation.

Transportation Data (49 CFR 172.101, .102)

DOT Shipping Name: Methyl alcohol
DOT Hazard Class: Flammable liquid
ID No.: UN1230
DOT Label: Flammable liquid
DOT Packaging Exceptions: 173.118
DOT Packaging Requirements: 173.119

IMO Shipping Name: Methanol
IMO Hazard Class: 3.2
ID No.: UN1230
IMO Label: Flammable Liquid, Poison
IMDG Packaging Group: II

MSDS Collection References: 26, 38, 73, 89, 100, 101, 103, 124, 126, 127, 132, 133, 136, 140, 143, 146, 148, 149, 153, 159, 163
Prepared by: M Gannon, BA; **Industrial Hygiene Review:** DJ Wilson, CIH; **Medical Review:** AC Darlington, MD, MPH; **Edited by:** JR Stuart, MS

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ATTACHMENT C-2

AIR MONITOR CALIBRATION INSTRUCTIONS

CUMMINGS RITER CONSULTANTS, INC.

EQUIPMENT CALIBRATION LOG

PROJECT NAME: _____

PROJECT NUMBER: _____

DATE: _____ Performed By: _____

INSTRUMENT TYPE: Hnu - Photoionization Detector - 10.2 eV Lamp

INSTRUMENT MODEL NUMBER: PI-101

INSTRUMENT SERIAL NUMBER: _____

DESCRIPTION OF CALIBRATION PROCEDURE:

a) Turn function switch to BATT to check battery.
b) Turn function switch to STANDBY - set dial to read zero with zero knob.
c) Connect the analyzer to the regulator and cylinder containing isobutylene/air mixture with a clean piece of tubing. (Do not use cylinder with < 30 psig.)
d) Turn the function switch to the 0-200 ppm range. Adjust the span to obtain calibration reading based on the concentration of isobutylene in the calibration gas. The concentration in the isobutylene of calibration gas is multiplied by 0.54 to obtain instrument reading. A calibration gas with an isobutylene concentration of 100 ppm provides on instrument reading of 54 ppm (i.e. 100 ppm x 0.54 = 54).
e) Recheck zero setting (Step B). If readjustment is necessary, repeat Step C.
f) Check operation and reaction with a permanent marker.

NOTE: Calibration was performed in atmospheric conditions similar to anticipated use area.

STANDARD pH OR CONCENTRATION	INSTRUMENT READING

REMARKS: _____

Span setting: _____

Background: _____

Calibration gas Lot #: _____

Calibration gas isobutylene concentration: _____ ppm.

ATTACHMENT C-3

MAP TO ST. JOSEPH'S HOSPITAL

Sorry! When printing directly from the browser your map may be incorrectly cropped. To print the entire map, try clicking the **"Printer-Friendly"** link at the top of your results page.

MAPQUEST.

START **1 Hardinge Dr**
Horseheads, NY 14845-2962, US

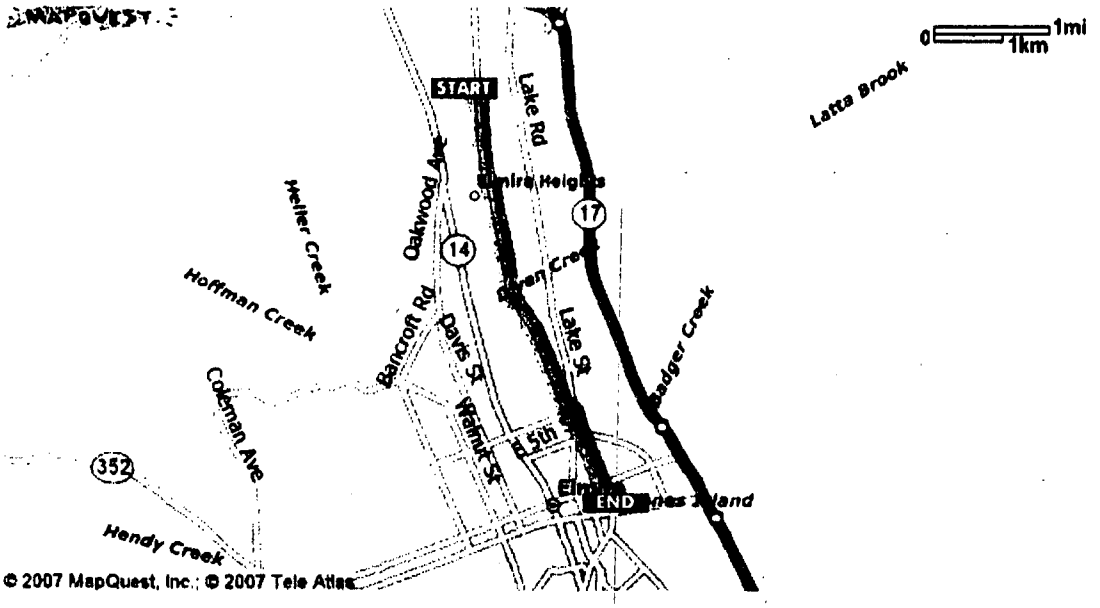
END **555 E Market St**
Elmira, NY 14901-3223, US

Total Est. Time:
10 minutes

Total Est. Distance:
3.98 miles

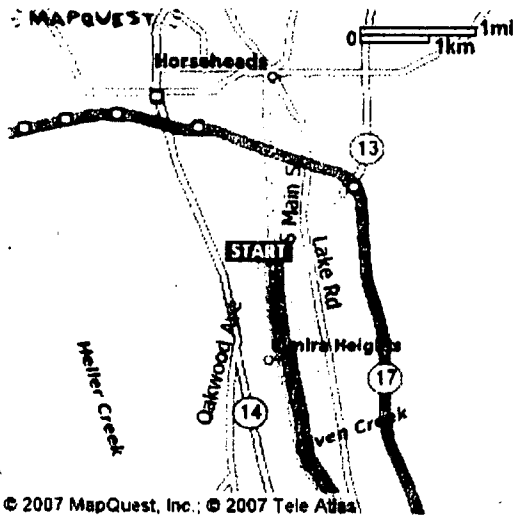
Maneuvers		Distance
START	**1:** Start out going EAST on OAKWOOD AVE / UPPER OAKWOOD AVE toward GRAND CENTRAL AVE.	<0.1 miles
	2: Turn RIGHT onto GRAND CENTRAL AVE.	2.9 miles
	3: Turn LEFT onto E WASHINGTON AVE.	<0.1 miles
	4: Turn RIGHT onto LAKE ST.	<0.1 miles
	5: LAKE ST becomes MADISON AVE.	0.7 miles
	6: Turn LEFT onto E MARKET ST.	<0.1 miles
END	**7:** End at **555 E Market St** Elmira, NY 14901-3223, US	

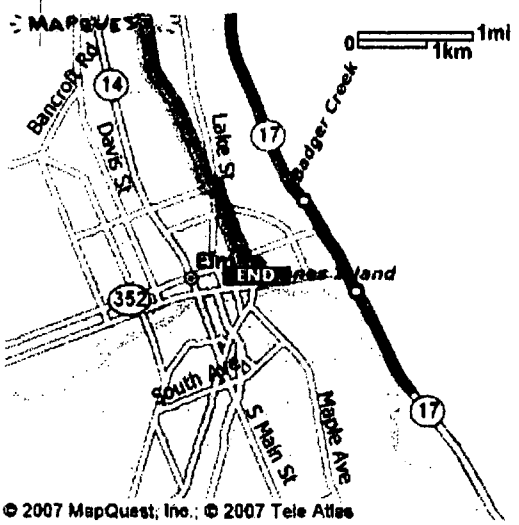
Total Est. Time: 10 minutes **Total Est. Distance:** 3.98 miles



Start:
1 Hardinge Dr
Horseheads, NY 14845-2962, US

End:
555 E Market St
Elmira, NY 14901-3223, US





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